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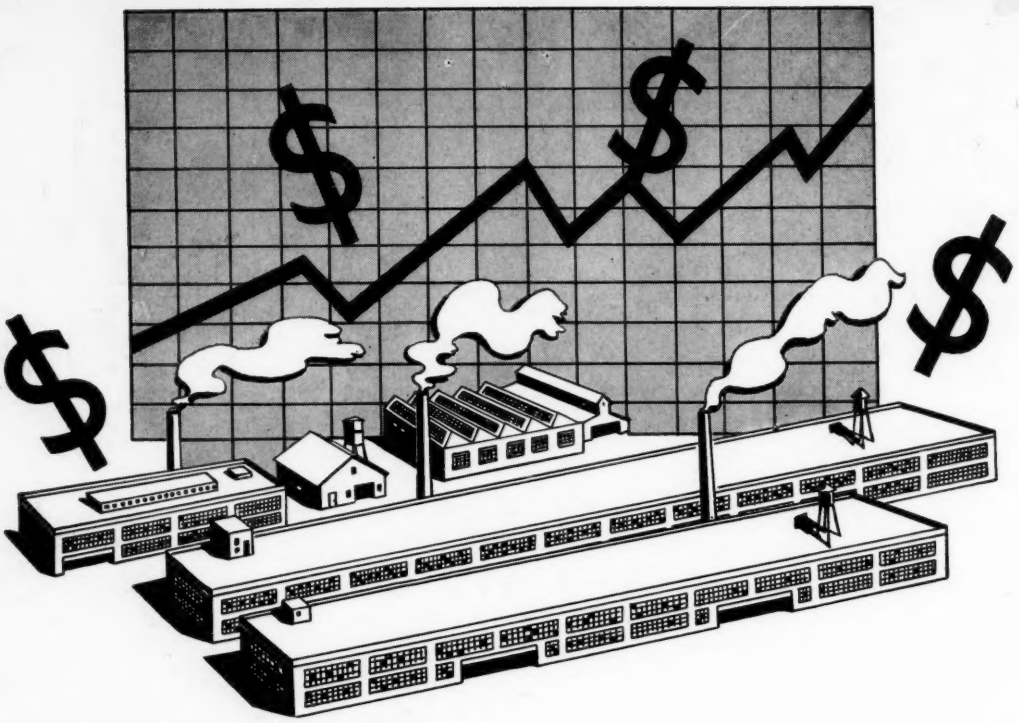

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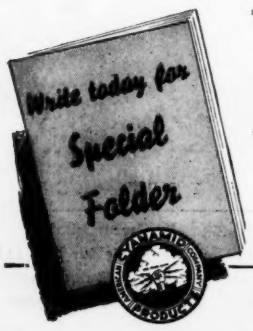

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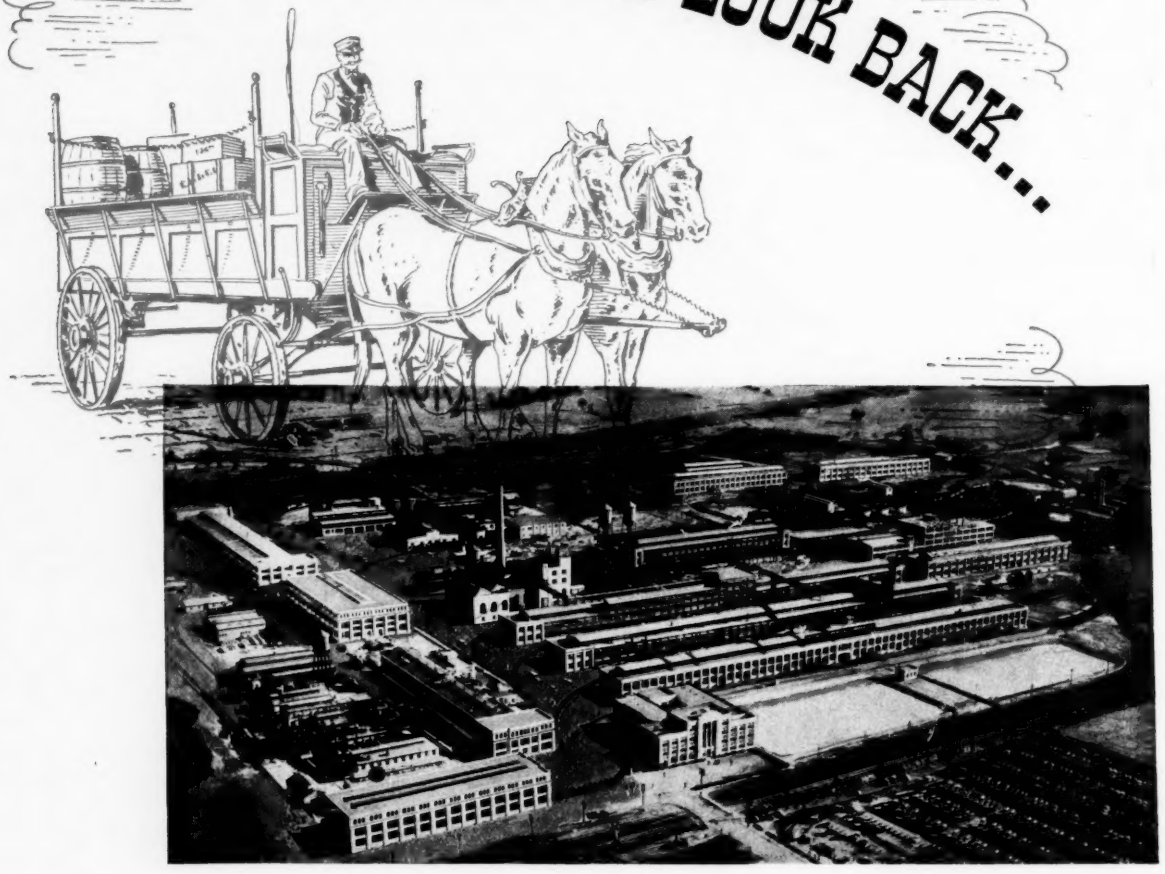

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VOLUME 30 JANUARY 20, 1941 NUMBER 2

Combination of Silk Fibroin with Acid and Base

LELAND F. GLEYSTEEN and MILTON HARRIS*

ABSTRACT

Silk contains a number of acidic and basic groups. The state of combination and the behavior of these groups are important in influencing the tensile and elastic properties of the fibroin and the adsorption of moisture and dyes.

As part of a broad research program of the Textile Foundation Research Associateship at the National Bureau of Standards on the acidic and basic properties of fibers, this investigation was undertaken to determine the relationship between the quantities of hydrochloric acid and potassium hydroxide taken up by fibroin from solutions and the concentrations of those solutions. The effect of added potassium chloride was also studied. In general, it has been possible to correlate the several parts of the titration curves of proteins with their amino acid contents, but our knowledge of the role of the phenolic hydroxyl groups of tyrosine has always been uncertain. Silk fibroin, however, contains such a large proportion of tyrosine as to practically assure an indication of the role of the phenolic hydroxyl groups of tyrosine simply by the magnitude of the base-binding capacity. In order to investigate more carefully the part taken by these groups, a titration curve was obtained for methylated fibroin in which most of the hydroxyl groups had been converted into non-reactive methoxyl groups.

The component parts of the titration curve of silk fibroin have been interpreted in terms of the composition of this protein. Thus the hydroxyl groups of tyrosine appear to bind base as shown by the fact that the base-combining capacity is much greater than can be accounted for by the other groups which are able to bind base.

The presence of free carboxyl groups in silk fibroin is suggested by the shape of the titraiton curve. From the maximum amount of acid bound an estimate of the total content of free amino groups may be obtained.

The effect of added salt is similar to that obtained with other insoluble proteins, namely to increase the quantity of acid or base bound at a given pH, in a manner which suggests that the combination of hydrogen ions is affected by the concentration of anions as well as by the concentration of hydrogen ions and that combination with hydroxyl ions is affected by the concentration of cations.

*Research Associates at the National Bureau of Standards, representing the Textile Foundation.

I. INTRODUCTION

MANY of the chemical and physical properties of a protein fiber are intimately related to the number and arrangement of those groups which bind acid or base. A study of the combination of acid and of base with silk fibroin was therefore undertaken as part of an extensive program for the investigation of the acidic and basic nature of textile fibers.

The manner in which the titration curves of the insoluble proteins, and especially of wool, differ from those of the soluble proteins has been discussed by Speakman and Hirst[1], by Lloyd and Bidder[2] and, more recently, by Steinhardt and Harris[3]. The titration of silk fibroin is, in some respects, similar to the titration of wool, but differs in other respects because of decided differences in the number and kinds of constituent amino acid residues. The published analyses of silk[4] indicate that fibroin contains relatively small quantities of the dibasic amino acids arginine, lysine and histidine, all of which would be expected to bind acid, and an appreciable quantity of one diacidic amino acid, tyrosine, which would be expected to bind base unless its phenolic hydroxyl group is blocked in some way.

The data for proteins do not provide an unequivocal indication of the availability of the hydroxyl groups of tyrosine for binding base. Recent work, however, on the reaction of diazomethane with silk and a number of other proteins indicates that the hydroxyl groups of tyrosine in these substances are probably free.

The relatively high content of tyrosine in fibroin made it seem probable that the role of the hydroxyl groups in

acid-base equilibria would be revealed by consideration of the titration data. Since the methylation of these groups can now be readily brought about[5], a comparison of the titration curves of untreated and of methylated fibroin should indicate even more clearly the part taken by the phenolic hydroxyl groups in the binding of base by fibroin.

II. EXPERIMENTAL PROCEDURE

1. Materials

(a) *Fibroin*

Raw white Japanese silk which had not been previously treated except for the reeling operation was extracted for 48 hours with alcohol and for 24 hours with ether, both at room temperature, and was finally washed in water. The silk was degummed by treatment with a commercial enzyme preparation, Enzo.* The degumming bath contained 5 g. of Enzo per liter and was maintained at a temperature of 65° C. The ratio of silk to solution was about 1:40. Each lot of silk was given three consecutive treatments of half an hour each with constant agitation; after each treatment the silk was quickly rinsed in boiling water and was then thoroughly washed in water at room temperature. In order to assure complete degumming it was found necessary to prevent the silk from becoming tightly matted. Determinations of loss of weight showed that three treatments of half an hour each and subsequent washings were sufficient to bring the material to constant weight. The loss of weight on degumming corresponded to the loss reported by Rutherford and Harris[6] for silk for the same lot degummed by boiling in soap solutions.

(b) *Methylated Fibroin*

Fibroin was methylated with diazomethane as described by Rutherford, Patterson and Harris[5]. Analysis of the samples for tyrosine by the method of Lugg[7] showed that the tyrosine content had been reduced from 12.0 to 2.3 per cent, and analysis for methoxyl groups by the method of Vieböch and Brecker[8] showed that the methoxyl content was 2.37 per cent.

2. Methods

(a) *Correction for Ash*

The degummed silk contained some base, already bound, and yields an alkaline ash. This alkali affects the extent to which acid or base is sorbed from the solution by the fibroin. The hydrogen ion equivalence of the ash was determined by an electrodialytic method which has been described elsewhere[9]. The degummed and washed fibroin had an ash content of the order of 0.1 milliequivalent per gram (*M*—eq./g.). In order to reduce the ash content to a lower value the fibroin was electrodialyzed for twenty-four hours. By this means the hydrogen ion equivalence of the ash was reduced to 0.01-0.02 *M*—eq./g. The titration data were corrected for the effect of this residual ash by subtracting the hydrogen ion equivalence of the ash from the experimentally determined quantity of acid bound, or adding it to the quantity of base bound.

*Thanks are due to the Wallerstein Laboratories, New York, N. Y., for the enzyme preparation used in this work.

(b) *Determination of Moisture*

All the samples were conditioned at 21° C. and 65 per cent relative humidity before weighing. The moisture content of the fibers was determined by drying representative samples for 2 hours in a vacuum oven at 105° C. All titration data in this paper are expressed as milliequivalents of acid or of base per gram of dry fibroin.

(c) *Correction for Sorption of Water*

In the determination of the titration curves of hygroscopic fibrous materials some investigators have considered the total moisture content of the sample of fibers, at the moment immediately preceding immersion in the solution, as a quantity which should be added to the free water of the system. That is to say, all the water in the system, including that introduced in the fiber phase, has sometimes been regarded as being available for dilution of the solute. However, many workers have recognized, on the basis of direct experimental evidence, that the concentration of an aqueous solution of a solute which is not bound to the fiber by strong chemical or physical forces is *increased* when a sample of fibers of low moisture content is introduced into the solution. Presumably such an increase in the concentration of the solution is due to the selective sorption of water by the fibers. It is also assumed that such sorption effects occur in solutions of acids and bases and decrease the quantity of water which is available for dilution of the acid or base. Since the acid and base binding capacities, as determined in the present investigation, are calculated from changes of titer and since the change in the titer of the solutions will depend upon the selective sorption of water as well as upon the quantity of acid or base bound a correction must be applied for the sorption of water.

The manner of applying this correction term is discussed in detail by Sookne and Harris[9a]. In the present investigation the extent of selective sorption of water was investigated by a polarimetric method. The highly optically active disaccharide trehalose was used as a reference substance. It was assumed that the trehalose was not sorbed by the fiber. A weighed quantity, 5 g. of fibroin of known moisture content was immersed in the smallest convenient volume, 60 ml. of trehalose solution, which was 0.05 *N* with respect to hydrochloric acid, and then placed in the constant temperature bath at 0° C. After 24 hours, aliquots were withdrawn from the stock solution and from the solution in equilibrium with the fibers and the rotations were measured in a saccharimeter at 20° C. The rotations of the original and of the final solutions were compared and the difference was interpreted as a measure of the increase of the concentration of the solution with respect to trehalose resulting from the sorption of water by the fibroin. The mean result for a pair of duplicate determinations is shown in Table I.

(d) *Titration with Acid*

Samples of fibroin weighing either 3.00 or 4.00 g. and containing about 9.2 per cent of moisture were placed in 100-ml. portions of acid solutions at 0° C. The solutions

TABLE I
Selective Sorption of Water by Silk Fibroin

Dry weight of fibroin	4.57 g.
Moisture content per gram of dry fibroin	0.095 g.
Change in rotation of solution	1.26%
Original volume of solution	60.0 ml.
Water selectivity sorbed	0.756 g.
Water selectively sorbed per gram of dry fibroin	0.166 g.
Total water in fiber not available for dilution of solute (grams per gram dry weight)	0.26 g.

contained either varying amounts of hydrochloric acid alone or varying amounts of potassium chloride and hydrochloric acid so adjusted as to bring the total ionic strength, at equilibrium, to 0.2 *M*. Aliquots of the original solutions and of the solutions which had come to equilibrium with the fibroin were titrated with standard sodium hydroxide solutions, using bromcresol purple as the indicator. The change in titer together with the dry weight of the sample and the correction for selective sorption of water permits the calculation of the quantity of acid bound per gram of dry fibroin.

To determine whether sufficient time had been allowed for the attainment of equilibrium and whether appreciable acid hydrolysis had occurred, aliquots were withdrawn from the acid solutions which had been in contact with the fibroin for periods of 2, 3 and 4 days. The results indicate that equilibrium is attained within 48 hours and that no appreciable hydrolysis occurs within four days at 0° C. in solutions up to 0.1 *N* with respect to hydrochloric acid.

(*e*) *Titration with Base*

Titrations with base were carried out in a manner analogous to the method used for acid titrations, with the addition of precautions to prevent errors resulting from exposure of the solutions to atmospheres containing carbon dioxide. The alkaline solutions were made up from a stock solution of potassium hydroxide of very low carbonate content and a stock solution of potassium chloride of very low carbon dioxide content, by dilution with freshly boiled distilled water.

To determine the time required to attain equilibrium and to investigate the question of the alkaline hydrolysis of the protein material, a number of samples were immersed in potassium hydroxide solutions at pH 12 for various periods of time. The results indicate that equilibrium is attained in about 24 hours at 0° C. and that no appreciable hydrolysis seems to have occurred within 72 hours at this temperature, as the quantity of base bound reached a constant value and the Nessler test did not reveal the presence of any nitrogen in solution. In all subsequent experiments the samples were allowed to remain in the solution for 48 hours before aliquots were withdrawn for titration.

(*f*) *Determination of pH*

The pH values of the solutions were determined with a glass electrode following the method used by Steinhardt and Harris[3].

(*g*) *Temperature Control*

The solutions were maintained at a temperature of 0° C. by immersion in a refrigerated bath containing water and ice.

III. RESULTS AND DISCUSSION

The amounts of hydrochloric acid and of potassium hydroxide bound by fibroin at 0° C. at different pH values (at equilibrium) are given in Table II and are shown graphically in Figure 1. The titration curve for

TABLE II.
Amounts of Acid and Base Bound by Silk Fibroin at 0° C.

No salt present

pH	*Acid Bound M—eq./g.*	*pH*	*Base Bound M—eq./g.*
1.083	0.132	9.01	0.033
1.131	.130	9.63	.034
1.553	.117	10.40	.052
2.046	.091	12.18	.147
2.331	.074	12.43	.205
2.468	.044	12.76	.314
2.950	.031	13.10	.395
3.522	.003	13.61	.798
		14.25	.904

Ionic strength 0.20

pH	*Acid Bound M—eq./g.*	*pH*	*Base Bound M—eq./g.*
1.187	0.136	9.10	0.070
1.822	.119	10.61	.139
2.101	.115	11.44	.228
2.360	.092	11.80	.298
2.875	.080	12.24	.487
3.138	.077	12.44	.581
3.276	.057	12.89	.686
3.968	.049	13.48	.767
4.633	.001		

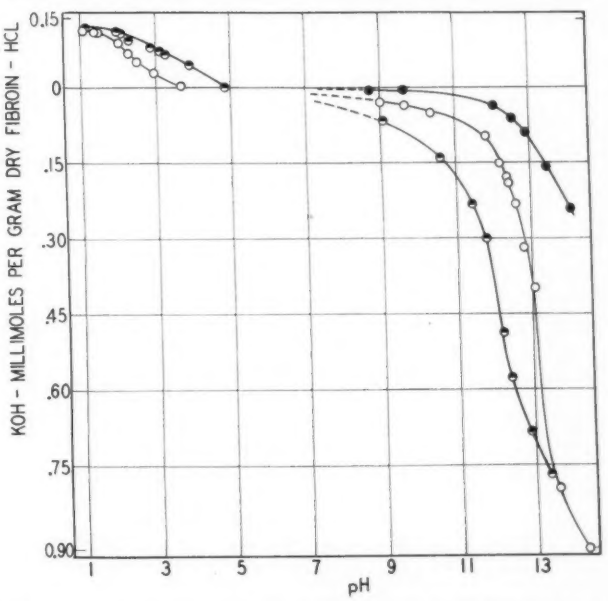

Figure 1. Titration curves of silk fibroin with hydrochloric acid and potassium hydroxide.
○ No salt
⊖ Ionic strength 0.2 *M*
● Methylated fibroin, no salt

hydrochloric acid in the absence of salt (open circles) drops steeply from a maximum of about 0.13 *M*—eq./g. near pH 1 and appears to reach the base line at about pH 3.9. It should be noted that becasue of the shallow slope

of the curve as it approaches the base line any error in the ash correction or in the titration data will affect the position of the point at which combination with acid appears to begin.

In solutions of chloride ionic strength 0.2 (half-filled circles) acid combination appears to begin at about pH 4.8. The effect of keeping the chloride ion at constant ionic strength 0.2 is clearly indicated by the figure. At the lower values of pH the curves tend to join each other, as indeed they must if the curve for constant ionic strength be extended to that point at which no salt is present. As in the case of wool[3], the central portion of both the acid and the alkaline regions of the curve for solutions of constant ionic strength is not as steep as the curve for solutions without salt, and a greater amount of acid or base is bound at a given pH. The effect of the presence of salt on the slope and position of the titration curve is greater than could reasonably be accounted for by the influence of the salt on the dissociation constants of the groups which are titrated. It will be observed that for the preservation of electrical neutrality in the solutions and in the fibers it is necessary that the fibers should bind an anion with every proton and a cation with every hydroxyl ion. A few determinations of the chloride titer of the acid solutions served to indicate that chloride ions are bound in quantities approximately equivalent to the amount of hydrogen ion bound. A detailed discussion of the effect of salt and a theoretical treatment of the titration data for another fibrous protein, wool, have been given elsewhere by Steinhardt and Harris[3], who showed that the quantity of acid bound depends upon the concentration of anions as well as on the concentration of hydrogen ions and that the quantity of base bound similarly depends on the concentration of cations as well as on hydroxyl ions. They explained the direct dependence on anion, or cation, concentration on the basis of the assumption that when the protein combines with hydrogen ions it also forms partially dissociated stoichiometric complexes with anions and when it combines with hydroxyl ions it forms similar complexes with cations. These conclusions now appear to be applicable to silk fibroin as well.

The titration curve for fibroin obtained from these experiments at 0° C. differs in several respects from that given by Lloyd and Bidder[2]. At the higher concentrations of acid Lloyd and Bidder indicate that rapidly and continuously increasing amounts of acid are bound as pH decreases, the highest value shown being some 0.3 *M*—eq./g. at pH 1; the data of the present investigation tend toward a maximum of about 0.13 *M*—eq./g. in the region of pH 1. This difference cannot be ascribed to the effects of ash because Lloyd and Bidder report an ash content of only 0.02 per cent which, on the assumption of an equivalent weight of 40 for the ash as found in the fibroin used in the present investigation, would amount to about 0.005 *M*—eq./g. The discrepancy may be due in part to some decomposition of the fibroin at the higher temperatures at which Lloyd and Bidder worked. The alkaline branches of the curves are less dissimilar and in this case at least a part of the difference may be attributed to a temperature effect. It has been shown that temperature appreciably influences the binding of base by wool but has very little effect on the binding of acid[11].

It is recognized that the acid- and base-combining capacities of proteins may usually be correlated with their contents of diacidic and dibasic amino acids[12]. The published analytical data on the composition of fibroin are given in Table III[4, 13]. The maximum quantity of acid

TABLE III
Amino Acid Composition of Fibroin

Amino Acid	*Per Cent*	*M—eq./g.*
1. Glycine[a]	43.8	5.84
2. Alanine[a]	26.4	2.97
3. Tyrosine[a]	13.2	0.73
4. Arginine[a]	0.95	0.055
5. Lysine[b]	0.25	0.018
6. Histidine[b]	0.07	0.005

[a]Reference [4].
[b]Reference [13].

bound by fibroin, 0.13 *M*—eq./g., is nearly twice the sum of the reported content of arginine and lysine. Such a discrepancy is not unexpected since in the isolation of small quantities of amino acids from protein hydrolysates the analytical results invariably are low.

In the literature no values whatever are reported for dicarboxylic acids in silk fibroin. The nature of the titration curve, however, is such as to suggest that dicarboxylic acid residues are probably present. The total amount of acid plus base bound between pH 1 and pH 7-8 (in which range the —COOH groups are known to undergo dissociation), which is not less than 0.13 and may be as much as 0.17 *M*—eq./g. in the case of silk fibroin, may be regarded as a measure of the total number of free carboxyl groups in the protein. Previous evidence that silk fibroin contains free carboxyl groups was given by Rutherford, Patterson and Harris[5], who infer the presence of about 0.2 *M*—eq./g. of free carboxyl groups in the untreated fibroin from the analyses of methylated fibroin for unmethylated tyrosine and for methoxyl groups. The latter estimate lends support to the interpretation of the quality of acid plus base bound between pH 1 and 8 as representing the free carboxyl content of fibroin.

The amount of base bound in the strongly alkaline region, that is at pH greater than 11, greatly exceeds the content of free carboxyl groups and can be accounted for only by postulating that the phenolic groups of the tyrosine take part in the binding of base. This result is not unexpected, as Rutherford, Patterson and Harris[5] concluded on the basis of their study of the methylation of fibroin with diazomethane that the hydroxyl groups are free. The greatest amount of base bound in the range covered by this investigation, 0.90 *M*—eq./g., is somewhat more than the tyrosine alone would account for and is, fortuitously, equal to the tyrosine content plus the estimated content of free carboxyl groups. Presumably higher amounts of

(Concluded on page 49)

Surface Characteristics of Cotton Fibers As Indicated by Electrophoretic Studies

ARNOLD M. SOOKNE and MILTON HARRIS*

ABSTRACT

Natural cotton is composed of a primary and a secondary wall. The former forms a thin sheath on the surface of the fiber and is composed principally of small amounts of cellulose imbedded in a membrane of wax and pectic substance. The latter is composed principally of cellulose and forms the main body of the fiber. The pectic substance, although present to the extent of only approximately 1 per cent, accounts for 85 per cent of the total acidic groups of the mature fiber. The highly acidic nature and the location of this substance in the naturally-occurring fibers would be expected to influence greatly a number of the properties of the fibers, especially those dependent on surface characteristics. These surface characteristics are of considerable importance in a number of industrial processes such as, for example, the scouring, dyeing, and finishing of textile materials.

Since the electrophoretic technique has been shown by research associates for the Textile Foundation at the National Bureau of Standards to be a useful tool for characterizing other fiber surfaces, it appeared advisable to apply it to an investigation of the cotton fiber as well. Samples of dewaxed cotton, cotton which had been depectinized by treatment with a boiling 1-per cent solution of sodium hydroxide for various lengths of time, and pectic substance from cotton gave widely different pH-mobility curves. The curve for pectic substance is characteristic of a highly acidic substance whereas that of depectinized cotton shows a low order of acidity. The curve for dewaxed cotton appears to be a composite of the curves for cellulose and pectic substance.

The purified cellulose exhibits a reversal of charge below pH 2.5, and accordingly is isoelectric at that pH. It is shown that the reversal of charge has not resulted from irreversible changes produced in the fiber during immersion in the dilute solutions of acid used in the present investigation.

I. INTRODUCTION

RECENT studies on cotton have indicated that the pectic substance occurs principally on the surface of the fiber as part of the primary cell wall[1, 2, 3]. Although this substance is present to the extent of only approximately 1 per cent, it nevertheless accounts for about 85 per cent of the acidic groups in mature fibers. The remainder of the acidic groups, about 15 per cent, are presumably associated with the cellulose itself[4]. The highly acidic nature of the location of the pectic substance in the naturally-occurring fibers, would be expected to influence greatly a number of the properties of the fiber, especially those dependent on surface characteristics. These surface characteristics are of considerable importance in a number of industrial processes, as for example, the scouring, dyeing and finishing of textile materials.

The electrophoretic technique has been shown in this laboratory to be a useful tool for characterizing and distinguishing surfaces of different textile fibers or of fibers of which the surface have been altered by different chemical treatments[5, 6]. Since it would be expected that the surface of a cotton fiber might also be appreciably changed by treatments which are known to remove pectic substance, it appeared that electrophoretic measurements would be of considerable aid in evaluating such changes.

II. MATERIALS AND METHODS

1. Materials

Raw cotton was extracted with alcohol at room temperature for 24 hours, and then with ether for 24 hours. This will henceforth be described as *dewaxed cotton.*

A portion of the dewaxed material was further purified by extraction for 2, 4, 8 or 16 hours with a boiling 1-per cent solution of sodium hydroxide, according to the method recommended for the preparation of standard cellulose. The procedure was essentially the same as that described by Corey and Gray[7], except that the apparatus of Worner and Mease was employed[8]. It has been shown by Whistler, Martin and Harris that this treatment removes all measurable amounts of pectic substance from cotton[9]. Samples purified in this way will be termed *depectinized cotton.*

All of the above samples were reduced to a convenient particle size by grinding them in a laboratory Wiley mill until they passed through a 60-mesh screen. The powder was suspended in water, the larger particles were allowed to settle, and the particles (1 to 5 microns) remaining in the supernatant liquid were used for the measurements.

Pectic substance was obtained by treating dewaxed cotton with a solution of sodium hexametaphosphate, shown elsewhere[10] to separate readily the pectic substance from cotton. The procedure was as follows: 100 g. of dewaxed cotton was soaked at room temperature for 7 hours

*Research Associates at the National Bureau of Standards representing the Textile Foundation.

in 1 liter of a 0.5-per cent solution of sodium hexametaphosphate at pH 7.9. The liquid was pressed out, and the cotton was soaked overnight in an additional 1-liter portion of the same solution. The combined solutions were dialyzed against running distilled water for 2 days. Hydrochloric acid and potassium chloride were added to portions of the solution until the final ionic strength was 0.02 *M* at pH values from 1.7 to 4.1. Microscopically visible particles suitable for mobility measurements were found to be suspended in the acidified solutions.

2. Methods

The electrophoretic technique was the same as that used in earlier studies on silk[5] and wool[6] fibers. An Abramson microelectrophoresis cell was used for the measurement of electric mobility[11]. The methods suggested by Moyer were followed in detail[12]. When the mobilities were very low, which was the case for all of the depectinized samples, the velocities of the particles were measured in successive layers from the top to the bottom of the cell, and the mobility was evaluated by graphical integration of the curve obtained by plotting velocity against depth[11].

Preliminary measurements showed that throughout the range of pH investigated, there was no appreciable change of the mobility of the pectic substance or of the dewaxed or depectinized cotton when suspensions were allowed to stand from 15 minutes to 24 hours before mobility measurements were made.

The pH values of the suspensions were measured with a McInnes and Belcher-type glass electrode and a vacuum-tube potentiometer, using a cathode-ray tube as null indicator. The pH values were referred to potassium acid phthalate, 0.05 *M*, to which was assigned a pH value of 4.01[13].

III. EXPERIMENTS AND DISCUSSION

Curve 5, Figure 1, shows the pH-mobility curve for pectic substance. The curve is typical of that of a highly acidic substance, since the particles exhibit a large negative charge even at pH 1.7. This is consistent with the results of an earlier investigation, which showed that the entire uronic acid-carboxyl content of the pectic substance was present in the form of titratable carboxyl groups[4]. The pH-mobility curve for dewaxed cotton (curve 4) is similar in shape to that for the pectic substance, but the material is shown by the shift in the curve along the axis of ordinates to be less acidic than the pectic substance. If the particles of dewaxed cotton were completely coated with pectic substance, a curve identical with curve 5 would have been obtained. The results suggest that cutting the fibers exposes new surfaces which are presumably cellulosic, and since these are much less acidic than the pectic substance itself, the number of acidic groups per unit surface has accordingly been decreased. It would appear then that curve 4 is a composite of the curves for the highly acidic substance and the much less acidic cellulose.

The pH-mobility curve of a sample of depectinized cotton. prepared by treating dewaxed cotton for 2 hours with a boiling 1-per cent solution of alkali, is shown in curve 3. Its position relative to the curve for dewaxed

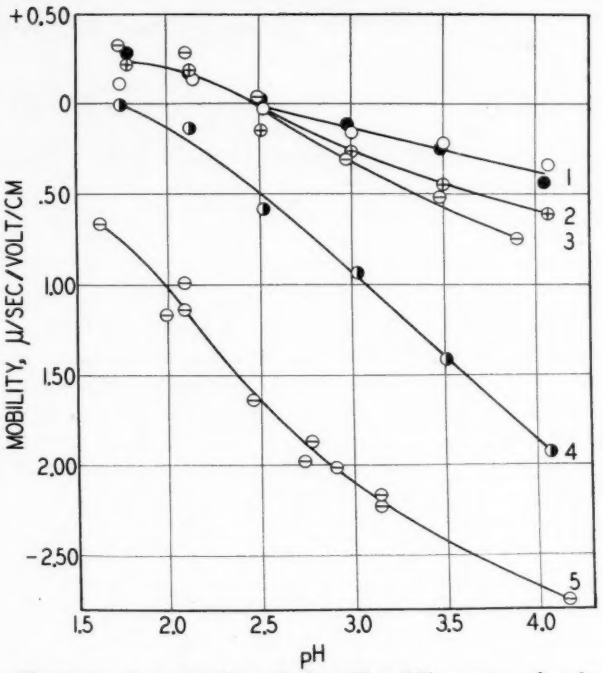

Figure 1. A comparison of the pH-mobility curves for dewaxed cotton, depectinized cotton, and pectic substance from cotton.

Curve 5 represents the mobility of the pectic substance. Curve 4 is that for dewaxed cotton. Curve 3 is that for cotton treated with boiling 1 per cent NaOH for 2 hours. Curve 2 is for cotton similarly treated for 4 hours. Curve 1 is for cotton treated for 8 (open circles) and for 16 hours (filled circles).

cotton indicates the removal of an acidic component. Slight further shifts in the pH-mobility curve were caused by 4 hours (curve 2) and 8 hours (curve 1) of extraction with the alkali, but these last effects are much smaller. A sample treated for 16 hours gave no further change in the pH-mobility curve (curve 1).

The small shifts in the positions of the pH-mobility curves for samples of cotton treated for 4 and for 8 hours were somewhat surprising, especially since earlier determinations of uronic acid[9] as well as titration studies[14] had indicated that essentially all of the pectic substance was removed from the fiber during the 2-hour treatment with alkali. It must be pointed out, however, that the electrophoretic measurements are much more sensitive to small changes than either of the above techniques and therefore the possibility that removal of the last traces of pectic substance during treatment with alkali for 2 hours is not complete, cannot be ruled out. If the data are interpreted in this light, then they must also be taken to indicate that the samples treated for 8 and for 16 hours are essentially free of pectic substance.

Two alternative explanations for these shifts may be advanced. First, the continued treatment with alkali may remove a small amount of surface material which contains no carboxyl groups but which is more electronegative than the cellulose itself. Second, the continued treatment with alkali may alter the physical structure of the fiber, with a

(Continued on page 49)

PROCEEDINGS OF THE

American Association of Textile Chemists and Colorists

JANUARY 20, 1941

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CALENDAR OF COMING EVENTS

Meeting, New York Section, Swiss Chalet, Rochelle Park, N. J., January 24, 1941.
Meeting, South Central Section, Read House, Chattanooga, Tenn., February 8, 1941.
Spring Meeting, Piedmont Section, George Vanderbilt Hotel, Asheville, North Carolina, April 12, 1941.

Annual Meeting and Convention, Carolina Hotel, Pinehurst, N. C., Oct. 31, Nov. 1, 1941.

Cationic Auxiliaries*

DR. H. H. MOSHER**

WE do not have to go back more than twenty years to find that at that time textiles was essentially an empirical art. The methods used were based upon the results of practical experiences rather than scientific developments.

Secret processes, secret methods of securing some particular color shade or physical effect, were rife throughout the industry and often constituted the most cherished legacy that an ageing dyer could bequeath to his sons.

Finishing operations as we understand them today, were almost non-existant and involved merely submitting a dyed fabric to some type of mechanical treatment to remove wrinkles and other forms of physical imperfections. Chemical finishing auxiliaries were few and confined to such natural products as flours and starches, soaps and occasionally emulsified oils, epsom salts and talc.

Textile materials could be subdivided into two general classifications, namely: those designed for ornamental purposes and appeal to the aesthetic senses of the consumer and those produced for utilitarian purposes. The differentiation between the two was primarily due to the fibers employed in the construction of the material. Silk owed its appeal to the inherent beauty of the silk filament and was used almost entirely in the preparation of ornamental fabrics. Utilitarian textiles were constructed of either cotton or wool; the former for general utility purposes, the latter for warmth.

The World's War produced radical changes in textile fashions and requirements and its aftermath keen competition between the textile producing nations.

That universal cataclysm disseminated, temporarily at least, far greater prosperity among our less prosperous citizenry and a means of gratifying an innate desire for personal adornment. A large portion of our population were no longer satisfied with the utilitarian merchandise, the badge of proletarianism, for street and party apparel, but demanded silk, and silk became a feminine prerequisite.

With recessions in mass prosperity and a lowered buying capacity, manufacturers of quality textiles succeeded in retaining their expanded clientele by the creation of novelties and the production of less costly merchandise, that similated the drape, handle and appearance of more luxurious by the employment of sizes, weighting agents and other artifices.

These newer creations often demanded revised dyeing methods, the use of more potent wetting, penetrating and dye levelling agents and improved finishing auxiliaries.

Rayon, after earlier failures and vicissitudes began to meet with popular approval at about this time. Rayon was a chemist's product, reared in chemical laboratories under scientific control, and its scientists made use of every technical advance to perfect the operating conditions of their scientific achievement. Scientists were attracted by the rayon industries to a study of textile operations and the results achieved induced dye chemists and textile technicians to direct their research proclivities to dyeing and finishing requirements and textile processing evolved from an empirical art into a science.

Today, the skillful dyer and finisher in his conversion of greige goods into a finished product may employ, in addition to his colors, detergents, wetting and penetrating agents, color levellers, dye fixatives, and a miscellany of finishing agents. All of these auxiliaries, with the exception of certain finishing agents, are surface active compounds, and it is to these that much attention has been directed by chemists and organic synthesists.

It was early observed that a striking similarity existed between the chemicals used in the scientifically developed lubricating industry and these surface active compounds.

When a drop of a properly developed lubricating oil impinges against a metallic object, a rapid flow of the lubricant occurs and after a short interval, the entire metallic surface is sheathed within a thin tenacious film. If cracks and pores occur in the metal, the oil penetrates down these imperfections tending to pry the object apart at these points of blemish. If rust or extraneous impurities coat the surface, each particle entity, like the metal itself is completely enveloped with an oily film. The impurities no longer are directly attached to the metal itself, but are separated by oil molecules. In consequence, when mineral oil is applied to soiled and rusty metallic objects and subsequently wiped, that object is freed from its impurities and the metal acquires its typical clean, shiny appearance.

The film strength and tenacity of these films on metals is truly remarkable. Oiled metal parts can seldom be wiped clean from a proper lubricant. A film of several molecules in thickness persists and tends to protect that metal from subsequent oxidation. That same characteristic is more strikingly demonstrated in the case of an oiled shaft. The shaft does not (or should not) come

*Presented before the Midwest Section, Saturday, Oct. 5, 1940.
**Director of Textile Research, Onyx Oil & Chemical Company.

in direct contact with its box, but rotates on oil molecules precisely as if they were the balls of a ball bearing.

Mineral oil exhibits the same tendency to form tenacious films about any object that it can wet; the same tendency to lift and suspend soil and impurities, the same tendency to pierce and penetrate cracks and pores; the same tendency to protect against atmospheric agencies. In brief, it acts on metallic objects in much the same manner that we expect surface active substances to act in aqueous solutions. Mineral oils, unfortunately, are not soluble in water and hence are, in themselves, not suitable for use in ordinary textile processing.

Lubricants apparently, however, bear the basic chemical structures for the formation of surface active substances. They may be compared to the body of a fish.

The body of the fish has no powers of projection through water in itself. It is only after that body is equipped with a tail and body vibratory movements that a virile aquatic creature is produced.

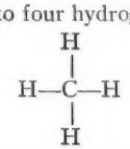
The lubricating oils constitute the body of surface active substances and it is incumbent upon the chemists and organic synthesists to attach the solubilizing tail and the driving power corresponding to the body movements of the fish.

CONSTITUTION OF MINERAL OILS

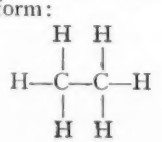
Most lubricating oils are derived from crude petroleum. Crude petroleum consists essentially of a complex mixture of hydrocarbons of different boiling points, which are roughly separated into different boiling fractions. The more volatile fractions are gaseous and receives industrial application as a fuel gas. Upon further fractional distillation, liquids of progressively greater viscosity and oiliness are secured, until finally a liquid distillate is produced that receives application as a lubricating oil.

If we should examine this fraction chemically, we would find that it, as does most of the petroleum fractions of crude oil of domestic production, consists of hydrocarbons of the methane series.

If we should examine all of the hydrocarbons, we would find that they group themselves into several homologous families. The most stable and most abundant as respects hydrocarbon members belong to a class of compounds known as the methane or aliphatic series. The simplest structural member is called methane and contains one carbon atom attached to four hydrogen atoms.



From methane, it is possible to yield a more complex homologue by replacing one hydrogen atom by a CH_3 radicle to form:



or C_2H_6

In a similar manner, a wholly homologous series of increasing complexity exists.

Formula	*Name*	*Melting Points*	*Boiling Points*	*Common Industrial Use*
CH_4	Methane	—186° C.	—160° C.	Fuel gases
C_2H_6	Ethane	—172° C.	— 93° C.	
C_3H_8	Propane	—156° C.	— 45° C.	
C_4H_{10}	Butane	—135° C.	— 0° C.	
C_5H_{12}	Bentane	—130° C.	36° C.	Solvent liquids, Petroleum ether, Ligroin, Naphtha, etc.
C_6H_{14}	Hexane	— 94° C.	68° C.	
C_7H_{16}	Heptane	— 97° C.	98° C.	
C_8H_{18}	Octane	— 56° C.	125° C.	
C_9H_{20}	Nonane	— 51° C.	150° C.	Fuels, gasolene, kerosene, etc.
$C_{10}H_{22}$	Decane	— 31° C.	173° C.	
$C_{11}H_{24}$	Undecane	— 26° C.	194° C.	
$C_{12}H_{26}$	Dodecane	— 12° C.	214° C.	
$C_{14}H_{30}$	Tetradecane	4° C.	252° C.	Lubrication oils Paraffin jellies waxes
$C_{16}H_{34}$	Hexadecane	18° C.	287° C.	
$C_{18}H_{38}$	Octadecane	24° C.	....	
$C_{20}H_{42}$	Eicosane	36° C.	....	
$C_{21}H_{44}$	Heneicosane	40° C.	....	

An examination of the table shows that each member of the series differs from the next simpler by a CH_2 group. It might be surmised that all members are much alike chemically.

Such is actually the case. A property possessed by methane is shared by every other member of the series though that characteristic is either acceleratingly accentuated or inhibited as we advance from the simpler to more complex family components.

The four simplest structures are gaseous and receive industrial application as gaseous fuels, but as we advance down the series to more intricate structures, we secure first light limpid liquids, that form weak filmy envelopments about metals and objects that they can wet. The viscosity, strength and tenacity of the film structures, and lubricity of the liquid increases with the larger molecules until we reach dodecane which is a true lubricating oil.

It may be classified more as a penetrant than a lubricant since its film-structures about metallic and other particles is usually not particularly strong.

Those hydrocarbons bearing between 14 and 18 carbon atoms possess the true lubricant structures, even though commercial lubricants are frequently vitiated with varying amounts of hydrocarbons bearing side chains and other complexities of structure.

The longer chained aliphatic members are jellies and solids and without value as ordinary commercial lubricants.

It is to the lubricating oil members of the aliphatic series that the attention of the chemist has been directed in the preparation of surface active auxiliaries. These yield the basic fat structures suitable for colloidal formations, suitable for the development of tenacious filmy structure about those colloids and materials that the hydrocarbon can wet. It is to these bases that chemists affix the solubilizing tails.

Such a tail should of necessity be easily wetted and soluble in water and provide that force or drive required for mobility of the resultant auxiliary in water.

The union or marriage of radicles of such divergent properties; the one water repellent and water insoluble; the other absorbing water with avidity and easily soluble, results in a compound, one end of which is fatty and water repellent, the other water absorbent.

The hydrocarbon moiety of such a product is called a hydrophobe, the solubilizing radicle a hydrophile.

The chemical synthesists, however, do not ordinarily employ mineral oils as the precursors of their textile auxiliaries. The members of the methane series are rather inert chemically and it is more convenient to use hydrocarbons to which hydrophile had already been attached. The fatty acids and saponifiable oils of nature constitute the raw product for most synthesis.

Nature was our first organic synthesist. It was she who first took the hydrocarbon and attached to its terminal ending, a solubilizing group, in her formation of the saponifiable oils and fats. These oils, however, were not elaborated for man's convenience but as necessary components of all life processes and, in themselves, are of but little value as surface active agents. Nature did, however, provide a hydrocarbon to which a hydrophile had been attached, but not the vital spark. It was encumbent upon man to free the fatty acid from its insolubilized glycerol ester and substitute a sodium radicle. That change provides the vital spark, the ion.

Oils and fats are the glycerol esters of various fatty acids. They are abundant all through nature and are essential ingredients of both animal and vegetable life processes.

They are insoluble in water, but are readily hydrolyzed by it, particularly in the presence of acids or alkalies to form fatty acids and, from these, soaps.

$$\begin{matrix} C_{15}H_{33}COO \\ \\ C_{15}H_{33}COO{-}C_3H_5 \\ \\ C_{15}H_{33}COO \end{matrix} + 3H_2O \rightarrow \begin{matrix} C_{15}H_{33}COOH \\ \\ C_{15}H_{33}COOH \\ \\ C_{15}H_{33}COOH \end{matrix} + {}_3H_5(OH)_3$$

Glycerol palmitate — Palmitic acid — Glycerol

$$C_{15}H_{33}COOH + NaOH \rightarrow C_{15}H_{33}COONa + H_2O$$

Palmitic acid — Caustic soda — Soap — Water

Soap was our earliest developed surface active material and, until a few decades ago, almost the only one that found application in industrial use. An explanation of its action and effect may be illustrative of every improved type of surface active material.

An inspection of the structural formulae of hexadecane and palmitic acid soap indicates that this soap may be considered as the hydorcarbon with its terminal CH_3 radicle replaced by a carboxyl group (COOH)

$CH_3(CH_2)_{14}CH_3$ — Tetradecane

$CH_3(CH_2)_{14}COONa$ — Palmitic acid soap

The soap is thus seen to be composed of a long chained structure, one end of which is insoluble in water and water repellent; the other is very easily soluble. The fatty moiety is called the soap hydrophobe, the carboxyl structure a hydrophile. Each terminal simultaneously tends to convey its inherent characteristic to the compound. The hydrophobe tends to dissolve fats and waxes, envelop all metallic and non-metallic colloids and surfaces that it can wet to form tenacious filmy sheathes and pierce and penetrate into all cracks and pores present on such objects. It likewise tends to render the film-enveloped objects water-repellent.

The hydrophile conversely tends to impart a very different effect. It tends to weaken film formations, to wet those substances to which its hydrophobic partner is attached and to inhibit or prevent penetration, but the hydrophile is inert without an electrical charge.

In aqueous solutions, almost all inorganic compounds and many organic are not present as molecules, but dissociate into ions, which are electrically charged particles. Molecules are in themselves electrically neutral, and in consequence, half of the ions bear a positive charge and the remainder a negative. The positive charged particles are called cations, the negatively charged, anions. Accordingly, a very dilute solution of hydrochloric acid is almost completely dissociated and is composed of positive charge H ions and negatively charge Cl.

$$HCl \rightleftarrows H^+ + Cl^-$$

Similarly, caustic soda solutions are essentially solutions of sodium cations and hydroxyl anions.

$$NaOH \rightleftarrows Na^+ + OH^-$$

When palmitic acid soap or sodium palmitate enters solution that liquor is composed not essentially of molecules, but of palmitic ions bearing negative charges and the cation sodium.

$$C_{15}H_{33}COONa \rightleftarrows C_{15}H_{33}COO^- + Na^+$$

It is that negative charge on the carboxyl ion that gives that radicle its driving force, that causes it to enter solution, to undergo chemical and even certain physical transformation.

Unlike charged electrical bodies always attract, like always repel, is one of the precepts of elementary physics. Most textile fibers, most sizes, finishes, and many classes of dye particles are negatively charged.

In consequence, the soap ions in aqueous solutions tend to be repelled by these fibrous structural orientations, colloids and ions.

Simultaneously, the soap hydrophobe tend to be adsorbed by the textile fibers and processing dyes and colloids so a balance exists between the two divergent forces.

The surface active properties of a textile auxiliary is a fine adjustment of these two forces. If either predominates to the exclusion or virtue extinction of the other, a valueless product results.

Soaps are not ideally constituted surface active bodies and the attention of textile technicians has been directed toward the securance of more potent hydrophiles for attachment to the long chained aliphatic hydrocarbon.

A great diversity of potentially available hydrophiles exists though the number in actual use are quite limited,

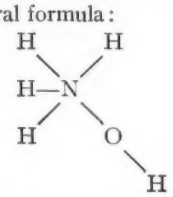
and most owe their solubility to the sulfate or sulfonate group.

All of the older surface active compounds and even many of the newest type are anion active. In consequence, the important part of the molecule, that moiety containing the fatty hydrophobe and its solubilizing hydrophile, bears in aqueous solution, a negative electric charge, and its properties are similar to those of soap, though often much more potent.

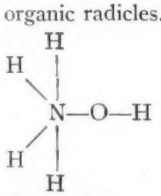
The past decade has witnessed the evolution of an entirely new type of surface active reagents, the so-called cation active auxiliaries.

These new compounds are mostly derivatives of nitrogen built around substituted ammonium hydroxide or the cyclic or heterocyclic nitrogen compounds such as pyridine.

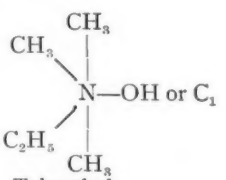
Ammonium hydroxide is a well-known chemical reagent bearing the structural formula:



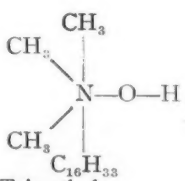
It is easily possible to replace the hydrogen atoms attached directly to the nitrogen atom with alkyl and other organic radicles.



Ammonium Hydroxide



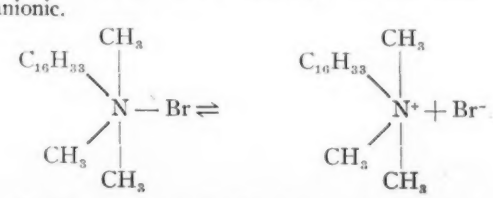
Trimethyl ethyl ammonium hydroxide



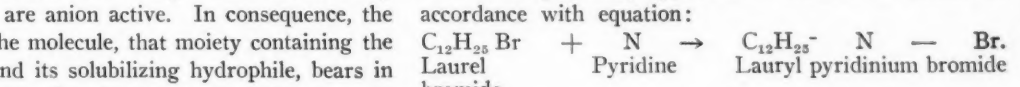
Trimethyl cetyl ammonium hydroxide

Derivatives of the type shown in the structural formulae above may be classified as substituted ammonium hydroxide and are usually called the quaternary ammonium compounds. Like ammonium hydroxide, these substituted derivatives are alkaline bases and may be neturalized with acids to form salts. If one of the substituted alkyl radicles is a long chained hydrocarbon radicle, the resulting compound is surface active. The hydrocarbon acts as the hydrophobe, the quaternary N as the hydrophile.

Such a product is unique among textile auxiliaries in that the active textile processing ion is cationic and not anionic.

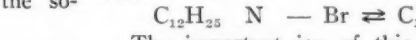


Cyclic and heterocyclic nitrogenous bases have been found useful as hydrophiles for long chained alkyl hydrophobes but to date only pyridine has received commercial utilization. When pyridine reacts with lauryl bromide, as an example, lauryl pyridinium bromide is formed in accordance with equation:



Laurel bromide — Pyridine — Lauryl pyridinium bromide

The lauryl pyridinium bromide like the corresponding quaternary ammonium salt is surface active and dissociates in water to yield a lauryl pyridinium cation and bromine ion.



The important ion of this auxiliary like that of the quaternary ammonium compound is cationic.

The cationic and older anionic textile auxiliaries exhibit striking similarities in structure and function and just as striking differences in behavior in solution.

Compounds of both types are long straight chained compounds with a hydrocarbon hydrophobe at one end of the molecule and a comparatively short hydrophile at the other. In each type of auxiliary conflicting physical forces are being exhibited; the hydrophobe tending to produce water-resistant tenacious films and penetrate any material that it can wet, whereas the hydrophile simultaneously seeks to neutralize or inhibit these characteristics. In each case, a proper balance of hydrophobic and hydrophilic forces is required for optimal surface active effectiveness. The balance may be varied to secure maximum wetting, penetrating, detergency or some desized element of surface activity.

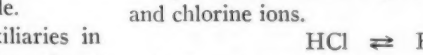
One characteristic difference between the two does exist. The long-chained quaternary ammonium and pyridinium auxiliaries in aqueous solution is a cation and bears a positive electrical charge;—the older type auxiliaries are anionic.

It is this positive charge that renders these compounds distinctive.

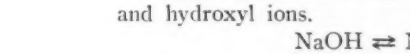
When two bodies bearing unlike electrical charges are present in the same bath, they migrate toward each other and where impingement occurs, the charges neutralize each other and a new chemical compound or aggregate results.

One of the simplest and most easily understood examples of this phenomenon is the neutralization of an acid by an alkali.

Hydrochloric acid in aqueous solution contains hydrogen and chlorine ions.



Sodium hydroxide in aqueous solution contains sodium and hydroxyl ions.



If the two solutions are mixed, the unlike charged ions are attracted to each other and temporarily unite. If the product formed is insoluble, volatile or non-ionizable, it is removed from solution. Hydrogen and hydroxyl ions unite to form water. Water is practically incapable of re-dissociation with the result that these ions are removed from solution and after evaporation sodium chloride results.

Almost all textile fibers, many textile auxiliaries and certain classes of dyestuffs are anionic (i.e., bear negative electrical charges.) As a consequence, when they are present in the same aqueous solutions as a quaternary ammonium auxiliary the two oppositely charged bodies migrate to each other, their charges neutralize at the points of impingement, and the radicles bearing the charges chemically unite to form new chemical compounds or new colloidal aggregates.

The significance of this phenomenon has been of tremendous importance to the textile industry and is receiving application in fabric softening, dyeing, finishing and in emulsion applications.

Many auxiliaries bearing fatty hydrophobes are softening agents. Most are anionic and but little substantivity between a textile and the softening agent exists. In fact, unless a softening emulsion breaks, there is actual repulsion. As a consequence, a textile seldom acquires a greater amount of an anionic softener from a finishing bath, than the concentration of that oil in the water adsorbed by the material being treated.

Relatively large amounts of a softening and plasticizing agent is required to produce potent results. Much of the auxiliary is distributed over the periphery of the filaments as a film and comparatively little is adsorbed into the intermicellar structure.

With cationic softeners, on the other hand, an entirely different condition exists. The fatty cation is attracted to the anionic radicles in the textile, ionic neutralization occurs and a more or less transitory chemical union results.

No haphazard films occur about fibers softened with cationic auxiliaries, but a true chemical orientation occurs. A quaternary ammonium auxiliary, as has been aforestated, is composed of two moieties, a long chained inactive hydrophobe, which imparts softness and a comparatively short reactive hydrophile bearing a positive electrical charge. The cnemical union between the fabric and softener occurs at the hydrophile, leaving the hydrophobic tail often extending out beyond the periphery of the filaments like a pin cushion full of pins, only in the case of the softener, the protruding barbs are soft and pliable.

As a consequence, a suitable cationic softener always imparts to fabrics a full, round, suede touch.

It is not to be inferred that all cationic bodies are necessarily superior softening agents. The hydrophobe must possess the requisite properties and the proper hydrophobic-hydrophilic balance must be secured.

Cationic auxiliaries are substantive and much of the chemical will be removed from a solution by a textile if sufficient time is allowed. From ten to twenty minutes is required for complete absorption. As a consequence, these softening agents are generally applied to textiles in the rinse bath subsequent to dyeing. The amount required to produce maximum efficiency is low and as a consequence, they have proven an inexpensive type of permanent softening agent.

The permanence of the chemical union between cationic softener and a textile fiber and the resistance of this union to laundering and drycleaning varies with the fiber to which the auxiliary is attached and to the particular softening agents. Many types of cationic softeners are possible. Some are as transitory as the older anionic and oil emulsion finishes, whereas some of the more potent and better balanced auxiliaries yield cellulosic complexes almost as permanent as the fiber itself.

Cationic softeners, however, directly possess but limited applicability in finishing operations. Softness is but one factor of finishing. Textile fabrics are of diverse construction and require great varieties of finishing effects. Simulations of additional body, firmness, weight and crispness are attributes of many textile finishing effects. Most of the finishing agents employed to impart these effects are anionic and hence inimical to use with cationic auxiliaries, the auxiliary reacting with the finish in much the same manner as with the textile, but in this case, the product of reaction is an insoluble conglomerate not serviceable for finishing purposes.

Indirectly, however, these cationic auxiliaries are valuable finishing adjuncts. The conventional types of fabric sizes, like the fabric itself, is often anionic. In consequence, but little ionic attraction exists between the fabric and size and whatever finish is acquired by the material is due to that substance contained in the moisture retained by the fabric after hydro-extraction. Where it is possible to reverse the electric charge on either the textile or the size to be applied, ready attraction between the two is secured.

An example of this method of finishing is the "rubberizing" of wool or silk by means of latex. The rubber colloids present in latex, like wool and silk, are anionic. But little affinity exists between the rubber and the textile.

Both of the animal fibers are amphoteric and as such, contain within their structures free groups, some of which are anion whereas others are cationic. The anionic character, however, predominates in alkaline solutions and latex particles are repelled rather than attracted to the textile.

If, however, the textile is treated with one of the cationic auxiliaries prior to its exposure to rubber a ready attraction is secured and wool or silk fabrics containing as much as 15 per cent rubber is easily secured. The explanation of the phenomenon depends upon the fact that cationic auxiliaries migrate to the free anionic groups of the textile neutralizing the negative electrical charges but leaving the positive charges intact. The animal fiber is thus converted into a cation fiber and, as such, possesses affinity for anionic bodies and colloids. Rubber colloids are anionic and, as such, migrate to the fiber and are deposited on and in its filament structures. Rubber so deposited may be considered as a direct chemical union with the textile and it thereby exhibits unusual resistance to laundering and drycleaning.

An alternative means of producing the same effect is to reverse the electrical charge on the rubber colloids in a latex solution. This is accomplished by the addition of a cationic auxiliary to the latex. The addition of an equivalent weight of the auxiliary to the latex results in complete precipitation of an electrical neutral colloidal agglutinate. A further addition produces dispersible rubber cationic complexes bearing positive electric charges.

These complexes, of course, being cationic, migrate to the anionic animal fibers and effective rubber sizes are thus secured on the wool or silk.

Other resins and sizing material can be applied in much the same manner.

This method of size application is still in its infancy but it has already demonstrated performance advantages. The size is, in most cases, firmly attached to the fabrics, often, in fact, in a direct though possibly weak pseudo chemical union. In consequence, a far firmer bond exists between the fabric and its finish. The finish is, of course, much more permanent, and less subject to "scratch marks" and cracking.

Although the cationic auxiliaries have proven a valuable finishing ingredient not only in its own right as a softening agent, but as an adjunct in reversing the electrical charges on either the textile fiber or on the colloidal size particles, their greatest textile application potentialities probably will be in conjunction with dyeing.

Most of the dye auxiliaries heretofore employed have been anionic surface active agents. Many of them foam vigorously, have potent wetting and penetrating properties and all tend to envelop dye particles in a filmy sheath. This sheath accentuates the anionic character of the dye colloids and hence inhibits its adsorptivity by textile fibers and produces a more gradual dye feed onto the fabrics being dyed. In brief, the auxiliaries act as dye retarders.

The cationic auxiliaries on the other hand are dyeing accelerators. They are as potent surface active agents as the anionic accelerators. The better balanced numbers foam as vigorously and wet out and penetrate as efficiently. They do, however, bear positive electrical charges and, as such, migrate to any negatively charged body present in a medium containing them forming colloidal aggregates or pseudo chemical complexes. In a dyebath both the dye colloid and textile bear negative charges, and in consequence, the cationic auxiliary will migrate to either or both.

Unfortunately, most of the aggregates which result from the union of the auxiliary and dye colloid are insoluble and the cationic surface active agents are not suitable for employment in the dye bath.

They may, however, be used in the bath after most of the dye has been exhausted as a substitute for Glauber's salt. The dye molecules, producing traces of color in this case, are quickly enveloped in a film of a cationic auxiliary such as the stearyl pyridinium ion and being positively charged, migrate to the fibers. The bath is quickly exhausted and those dye ions, that had prior to the addition of the auxiliary to the liquor, been affixed by the fibers being colored, united with the excess of auxiliary becoming less soluble, and the fabric is faster to both washing and drycleaning and more resistant to crocking.

Much better results, however, are experienced by reversing the electrical charges on the fabric prior to its addition to a dye bath, in a manner similar to that described in applying latex to silk or wool.

The fabric should first be boiled for a period of from twenty to thirty minutes, in a solution containing from six to eight per cent of a cationic auxiliary such as stearyl pyridinium bromide. Most of the auxiliary by this treatment has affixed itself to the textile and reversed its electrical charge. The newly acquired cationic properties of the textile permits of a rapid and uniform acquisition of color from a dye solution. The color secured in this way is much more permanent to washing and crocks less.

A process similar to that described is of particular interest in applying direct colors to cellulosic fabrics. These colors do not possess particularly good wash fastness on rayons and cottons, but are inexpensive and industrial economics often decree their use.

A preliminary treatment in the cationic auxiliaries frequently imparts to the dyes the requisite industrial fastness.

Such a preliminary treatment likewise permits of the application of the usual wool and wool mordant dyes to cellulosic fibers thus simplifying the dyeing of wool-rayon or wool-cotton unions.

Improved color fixation can also be secured by treating dyed fabrics with solutions of cationic auxiliaries. The mere passage of the fabric through such a solution results in a migration of the cationic auxiliary to the anionic fibers and affixed dye ions where insoluble aggregates are produced. The extreme insolubility of these dye auxiliary complexes prevent their solution in water and hence inhibit bleeding. The treated material is likewise resistant to perspiration. The washing of prints in solutions containing such cationic auxiliaries is thus becoming quite general. Lining cloths and other textiles subject to bleeding by perspiration are aided by applications of cationic auxiliaries subsequent to dyeing.

Surface active materials tend to exhibit potent wetting, penetrating, foaming, emulsifying and detergent properties. The efficiency of any one of these characteristics is dependent upon a variety of factors such as the length or nature of the hydrophobic or hydrophilic moiety and the balance between the two. In consequence we might expect to secure as potently potential cationic emulsifying agents and detergents as anionic.

Auxiliaries of extremely potent emulsifying and potential detergent properties have been prepared. Stearyl or cetyl pyridinium bromide are suitably balanced auxiliaries for these effects.

Either stearyl or cetyl pyridinium will emulsify oils and fats and produce a stable emulsion. Such emulsions to be of general industrial interest should be stable under

normal methods of applications. However, most materials to which these oil dispersions would be applied are anionic. Such materials have a selective affinity for the cationic disperser and, as this is removed from solution, the emulsion breaks and the oil or other materials dispersed, separate.

Such a condition is desired in the throwster's soaking bath where complete oil exhaustion is demanded. It would appear that the cationic auxiliaries will receive accelerating increased uses in this industry, as complete oil exhaustion is easily secured and as the auxiliaries themselves produce a potent softening and plasticizing action on the silk gum.

Although cationic detergents such as cetyl pyridinium bromide seem destined to never be a major factor in cleaning and scouring, because of their substantivity to the materials being processed, they are by no means devoid of interest to the textile chemist.

Wool, worsted and fur fibers in general are very susceptible to deterioration in alkaline solutions though very resistant to acids. A detergent effective in acid solutions has long been sought. Such a material would protect animal fibers against every potential damage during scouring and would also permit of the development of single bath scouring and dyeing operations for processing wool yarns and fabrics.

Wool in common with most textile fibers under alkaline or neutral conditions is anionic. However, wool is amphoteric and possesses both anionic and cationic radicles with the anionic predominating. When animal fibers react in solutions more acid than its iso-electric point, the charge reverses on the wool and it becomes, not anionic but, cationic, and in consequence can be cleaned with cationic detergents.

Contrary to other scouring agents, the cationic detergents exhibit their maximum effectiveness under conditions of acidity.

Since both the wool and cetyl pyridinium bromide bear the same electrical charges under these conditions, no wool substantivity for the scouring agent would result. Laboratory tests have yielded very promising results in the cleaning of animal fibers by these cationic detergents under acidic conditions and in the simultaneous dyeing and scouring of woolen materials under acidic conditions. The method to the best of my knowledge has not yet been put into industrial use. It is possible that a perfectly formulated cationic detergent has not yet been produced, but if not, we can confidently expect its synthesis in the near future.

One of the most unexpected properties of the cationic auxiliaries have been their antiseptic action. Most antiseptics are potent and corrosive poisons. Cationic auxiliaries, however, are but little more toxic than soap and as mild in their action on the skin as a fat alcohol sulfate. It has indeed been surprising to find such agents rivals to the mercury and the silver salt in bacteriocidal properties. Like most of its other salient and valuable characteristics that can be traced in part to its cationic nature. Most micro-organisms are anionic colloids. Naturally in the presence of the auxiliary migration of the one to the other occurs and some type of pseudo chemical reaction occurs. Just what happens after the neutralization of electro charge is complete are matters of speculation. It has been proposed that the hydrophobic moiety of the auxiliary wraps itself about the organism encasing it in a filmy tomb from which it is unable to secure its necessary supply of oxygen.

The development of permanent water proofing agents has been perfected through the use of cationic auxiliaries.

The advance of scientific textile processing has been amazingly rapid during the past few decades. Many paths lead to new vistas of achievement. The cationic auxiliaries are one such path. To what horizons this path may stretch and what achievements may be accomplished by the use of cationic auxiliaries, we can but speculate, but one thing we do know, the cationic auxiliaries are already useful tools in the hands of a skillful textile processor.

MEETING, RHODE ISLAND SECTION

THE Rhode Island Section met in the Providence Engineering Building on November 15, 1940, with attendance of one hundred and fifty. Charles F. E. Sedgwick, Superintendent of Preparation, Esmond Mills, spoke on, "Preparation for the Dyeing of Wool Raw Stock"; and Wallace Taylor, Sales Manager, The Textile Finishing Machinery Co., spoke on "Streamlining of Textile Dyeing and Finishing Machinery, and Present Day Needs."

Respectfully submitted,

LOUIS D. FLEMING, *Secretary.*

MEETING, NORTHERN NEW ENGLAND SECTION

THE Northern New England Section held its first meeting of the 1940-41 season on Friday, November 15, 1940, at the University Club, Boston, Massachusetts.

John N. Dalton, newly-elected chairman, presided over the meeting, which was attended by approximately one hundred and twenty members. In addition, there were a number of textile executives present, on special invitation.

Dinner was served at 6:30 p.m., and followed by a short business meeting.

The speaker for the evening was Lieutenant-Colonel Vere Painter from the Quartermaster's Department in Philadelphia, the officer in charge of the inspection of textiles. Lieutenant-Colonel Painter's subject was "National Defense and the Relation of the Textile Industry Thereto." At the conclusion of his talk, Lieutenant-Colonel Painter answered many questions and ironed out a good many problems that confronted our members engaged in filling government contracts for national defense.

The meeting concluded with remarks by Franklin W. Hobbs, President of the Arlington Mills and of the Textile Foundation, Inc.

Respectfully submitted,

EDWARD S. CHAPIN, *Secretary.*

Some Uses of Tetra Sodium Pyro Phosphate in the Textile Industry*

PAUL LOGUE**

HISTORICAL

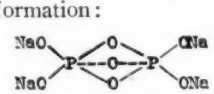
MORE than 100 years ago, in 1827 to be exact, Thomas Clark, a Scotchman of Edinburgh, made the first Tetra Sodium Pyro Phosphate by heating disodium ortho phosphate. He identified it as differing from his starting material by the observation that its aqueous solution gave a white precipitate with silver nitrate instead of the usual yellow precipitate of the ortho phosphate. It was 101 years after this discovery that the product first appeared in literature as of commercial interest in the form of a German patent issued in 1928.

It was five years prior to this, however, that our company produced Tetra Sodium Pyro Phosphate in ton lots, the first, so far as is known, of commercial production on this scale in the United States. There then followed an induction period of ten or twelve years before the advantages of Tetra Sodium Pyro Phosphate were appreciated by the consuming trade and widespread use became effective. Thus it appears that several generations of our ancestors went to their graves deprived of the benefits of this Scotchman's discovery and of the blessings which are ours today through the use of Tetra Sodium Pyro Phosphate.

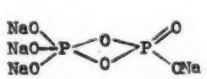
Following the good American custom of free competition, the price of Tetra Sodium Pyro Phosphate has declined in proportion to the increase in volume of consumption from an original cost of 16 cents per pound through successive reductions, until now it is available at $5.10 per hundred pounds in carloads, f.o.b. works, which places it among the lowest cost detergent compounds available on a 100 per cent active basis.

PHYSICAL PROPERTIES

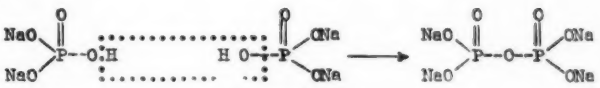
The empirical formula for Tetra Sodium Pyro Phosphate was established in 1833 by Graham as $Na_4P_2O_7$. Structurally it has been written as follows, although Formula III appears to be the most logical from its method of formation:



Formula I



Formula II



Formula III

Tetra Sodium Pyro Phosphate was shown by Partridge, Hicks and Smith in a paper read before the American Chemical Society last June to exist in five enantiotropic forms, but due to the complete reversibility of these inversions, samples with varying thermal histories all show the same X-ray pattern at room temperature. It melts at 985° C., crystallizes from water solutions as $Na_4P_2O_7.10\ H_2O$, and a 1 per cent aqueous solution of the anhydrous salt has a pH of 10.2. The pure compound contains, theoretically, 53.40 per cent P_2O_5 and 46.60 per cent Na_2O. Commercially available products run 98 to 99.5 per cent Tetra Sodium Pyro Phosphate. Solubility data are shown in Figure II and pH values in Table I.

TABLE I
pH of 1 Per Cent Solutions of Commonly Used Chemicals

Caustic Soda	13.3
Sodium Meta Silicate	12.4
Tri Sodium Phosphate Crystalline	12.1
Soda Ash	11.5
Tetra Sodium Pyro Phosphate	10.2
Borax	9.0
Di Sodium Phosphate Crystalline	8.9
Sodium Bicarbonate	8.3
Mono Sodium Phosphate Anhydrous	4.6
Sodium Acid Pyro Phosphate	4.2

The value of Tetra Sodium Pyro Phosphate rests upon its ability to soften water and peptize finely divided solids at mild alkalinity or low pH. The pH of useful concentrations of Tetra Sodium Pyro Phosphate is no greater than that of neutral soap. Its greatest commercial application as a soap builder in household package soaps is primarily because of these characteristics.

The water softening power of Tetra Sodium Pyro Phosphate is shown in Figure I. As a measure of the peptizing characteristics of Tetra Sodium Pyro Phosphate, the number of milligrams of burnt umber remaining in 100 ml. of suspension 20 hours after the suspension is first formed is shown in accompanying Figure IV.

Another characteristic of Tetra Sodium Pyro Phosphate having an important bearing on its usefulness in the textile industry is its ability to solubilize iron salts. It forms complex sodium iron pyro phosphates, having much greater solubility in water than the simple ferrous or ferric pyro phosphates themselves.

*Presented at Symposium on Chemical and Dyeing Aids for the Textile Industry, Annual Meeting, New York, N. Y., October 19, 1940.

**Monsanto Chemical Company, St. Louis, Missouri.

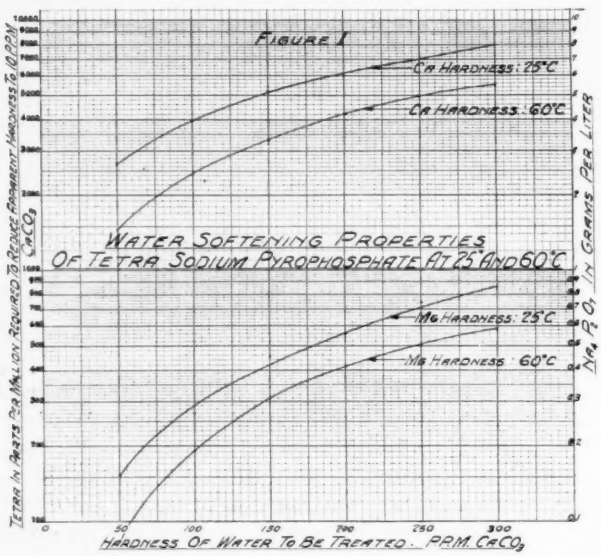


FIGURE I
WATER SOFTENING PROPERTIES
OF TETRA SODIUM PYROPHOSPHATE AT 25 AND 60°C
Ca HARDNESS: 25°C
Ca HARDNESS: 60°C
Mg HARDNESS: 25°C
Mg HARDNESS: 60°C
HARDNESS OF WATER TO BE TREATED: P.P.M. CaCO3
Na4P2O7 IN GRAMS PER LITER

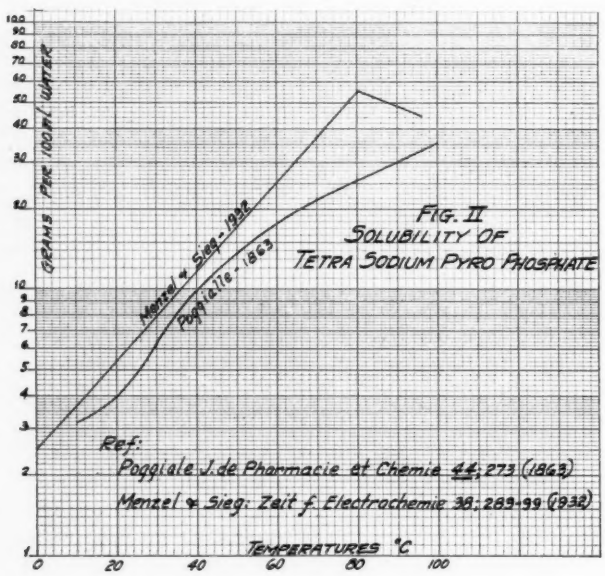
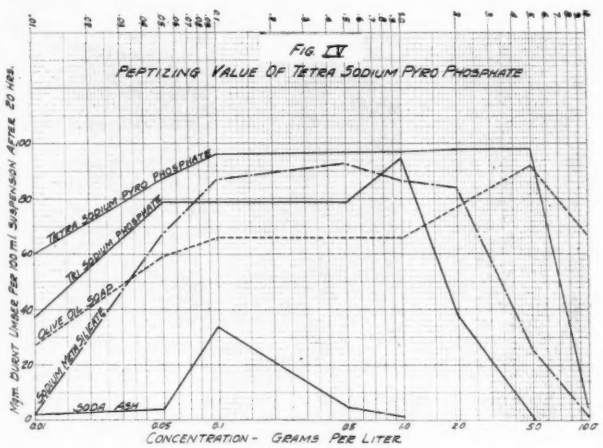


FIG. II
SOLUBILITY OF
TETRA SODIUM PYRO PHOSPHATE
Menzel & Sieg - 1932
Poggiale - 1863
GRAMS PER 100 ML WATER
TEMPERATURES °C
Ref:
Poggiale J. de Pharmacie et Chemie 44; 273 (1863)
Menzel & Sieg: Zeit f. Electrochemie 38; 289-99 (1932)



APPLICATIONS

For specific applications of the Tetra Sodium Pyro Phosphate to the textile industry we may begin with cotton, and the first chemical step in cotton processing is usually kier boiling. Tetra Sodium Pyro Phosphate is especially useful in this operation where a soft absorbent finish is desired.

The data shown in Table II covers tests on 36" cotton sheeting of the following characteristics:

Warp wise tensile strength.....	57.6 pounds
Filling wise tensile strength.....	61.6 pounds
Texture	64.5 x 69.5
Weight (yards per pound).....	3.41

Grey goods, previously desized, rinsed and dried, were processed in a closed kier for 6 hours at 250° F. and a pressure of 15 pounds per square inch. Following kiering treatment, the cloth was rinsed thoroughly and dried at approxmiately 100-105° F.

One-yard test pieces were conditioned overnight at 70° F. and 65 per cent R.H. and then tested and examined as outlined below:

1. Shrinkage and contraction. Standard A.S.T.M. Method.
2. Loss in weight. Calculated on basis of original conditioned weight of grey goods.
3. Texture. Pick counter.

TABLE II
Quantitative Data on Kier Boiling Cotton Sheeting

	Kiering Formula			*Water*	*Shrink-*	*Contrac-*	*Loss*	*Tensile*	*Color*	*Absor-*
	Caustic Soda %	*TSPP* %*	*Sodium Silicate %*	*Hardness ppm.*	*age %*	*tion %*	*in Wt. %*	*Strength % Orig.*		*bency (Seconds)*
1	3.0	1.0	...	90	9.3	7.6	13.4	93.00	+	13
2	3.0	1.0	2.0	90	9.0	7.3	11.3	93.75	+	17
3	3.0	...	...	Zero	8.0	5.8	11.2	92.00	S	18
4	1.5	1.5	...	90	8.3	6.6	11.5	90.65	+	34
5	3.0	0.5	...	90	8.6	7.9	11.1	98.25	+	36
6	3.0	...	2.0	90	8.6	6.9	11.5	82.60	+	48
7	2.0	1.0	...	Zero	9.6	8.0	12.8	89.35	+	75
8	2.0	1.0	...	90	9.2	6.8	12.7	29.35	+	80
9	3.0	...	...	90	9.0	7.5	11.6	89.05	S	>1800
10	5.0	...	...	90	8.9	7.6	13.0	85.70	..	>1800

4. Tensile strengths. Average of 5 or more breaks, both warp and filling, by standard grab method on Scott Testing Machine. Results were corrected for shrinkage on basis of change in texture, and are expressed as per cent of original grey goods breaking strengths, averaged as to warp and filling.
5. Absorbency. Comparative time in seconds required for 2" discs to wet out and sink in a beaker of water.
6. Color. Comparative appearance, i.e., whiteness and freedom from brownish stains. Results expressed as: S, equal to standard 3 per cent caustic soda formula; —, worse than standard; and +, better than standard.

Attention is directed to the high absorbency (low wetting time), good color and tensile strength of tests involving Tetra Sodium Pyro Phosphate. These results may be summarized as follows:

1. In soft water and using approximately 3 per cent total kiering agents. Improved color and elimination of brownish stains.
2. In hard water and using approximately 3 per cent total kiering agents. Improved color and elimination of brownish stains. Increased absorbency.
3. In hard water and using more than 3 per cent total kiering agents.
   a. No silicate present:
      Improved appearance and greater absorbency as above.
      Increase in tensile strength. Approximately 8 per cent.
   b. With silicate also present:
      Appearance improved and greater absorbency as above.
      Increased tensile strength. Approximately 7-12 per cent.

Some typical commercial examples follow:

1. Type of Goods: Single and ply yarn and cables.
   Water: 90 ppm. hardness.
   Conditions: Boil 3 hours at 214° F.

Formulae:	*Old*	*New*
Caustic Soda	1.6%	1.6%
Sodium Silicate	3.84%	3.84%
T.S.P.P.*	none	0.8%
Wetting Agent	1.6%	1.6%

   Results: Marked reduction in loss of tensile strength. Goods kiered with T.S.P.P. had tensile strengths 7-12 per cent higher than when old formula used.
2. Type of goods: Woven piece goods—flannels, sheetings, duck, etc.
   Water: 80 ppm. hardness.
   Conditions: Boil 14 hours at 25 lbs. pressure.

Formulae:	*Old*	*New*
Caustic Soda	3.12%	4.0%
Soda Ash	3.75%	none
Sodium Silicate	1.5%	1.5%
T.S.P.	1.0%	none
T.S.P.P.*	none	1.0%
Wetting Agent	0.075%	0.075%

   Results: Chemical costs reduced without affecting quality. Caustic was available as waste from mercerizing unit.
3. Type of Goods: Turkish towelling.
   Water: 18 ppm. hardness.
   Conditions: Not given.

Formulae:	*Old*	*New*
Caustic Soda	3%	3%
Soda Ash	1%	none
Sodium Silicate	1.5%	1%
T.S.P.P.*	none	1%
Wetting Agent	small amount	small amount

   Results: Finished goods were softer and more absorbent, which was very desirable for type of products processed.
4. Type of Goods: Light weight woven goods.
   Water: 100 ppm. hardness.
   Conditions: Not given.

Formulae:	*New*
Caustic Soda	3.0%
Sodium Silicate	0.25%
T.S.P.P.*	0.5%

   Results: Gets cleaner kier and eliminates resist spots in dyeing, which are due to insoluble calcium soaps formed in the kier.

BLEACHING

While under most conditions of operation Tetra Sodium Pyro Phosphate has not been found to be as good as sodium silicate in stabilizing the bleach bath, it has, nevertheless, enjoyed a certain usefulness in bleaching operations, especially where hydrogen peroxide is used. The property believed to be of especial value in this connection is its ability to form soluble iron and calcium compounds on the alkaline side.

A couple of examples of commercial practice will serve to illustrate the specific usefulness of Tetra Sodium Pyro Phosphate in the field of application.

1. A manufacturer of diaper cloth uses a calcium containing sizing compound in sizing the warp to assist in weaving. This is not thoroughly removed in the kier boiling and causes resist spots when the cloth is tinted. He has not been able to effect complete removal of the lime by adding Tetra Sodium Pyro Phosphate to the kier (probably due to the abnormally high pH) but finds that by adding it at the rate of 0.5 per cent to the hydrogen peroxide bleach bath at the beginning of the operation, completely satisfactory results are obtained.

2. Another manufacturer uses 1 per cent T.S.P.P.* on the weight of the dry yarn in his package bleaching machine, added five to ten minutes before the operation is complete. This prevents iron stains, which he previously encountered to a serious degree. After several weeks' use he found a thin, white coating over the inner surface of the iron machine which acts as a protective lining. In his case he found it impossible to add the Tetra Sodium Pyro Phosphate at the beginning of the bleaching operation because when he tried this the evolution of hydrogen peroxide was so rapid the machine "blew." This is believed to have been caused by bronze heating tubes in the heat exchanger.

DYEING

Naphthol Colors are attracting more and more attention because of their excellent washing, bleaching and light fastness, and also because they are relatively cheap to apply. The principal disadvantage of the naphthol dyes lies in the fact that they tend to crock (smudge contiguous white fabric areas). It has been found that addition of Tetra Sodium Pyro Phosphate to the soap used in "soaping off" after the dyeing is complete effectively loosens and removes the unabsorbed and precipitated dyestuff adhering to the surface of the fibers and results in cleaner yarn and brighter shades with much less tendency to crock. The optimum ratio appears to be 20-25 per cent of Tetra Sodium Pyro Phosphate, based upon the weight of the neutral soap employed. A customary commercial formula consists of 5 per cent soap and 1¼ per cent Tetra Sodium Pyro Phosphate, based upon the weight of the dry yarn or cloth.

Direct Colors. A dyer found difficulty with a direct green crocking off on the leather covers of his spinning rolls, resulting in contamination of subsequent lighter shades passing through the same machine. By adding 1 per cent Tetra Sodium Pyro Phosphate (on the weight of dry cotton) into the dye solution as it was boiled up before pouring into the dye machine, this crocking trouble was eliminated.

T.S.P.P.* is used in both raw stock and package dyeing of various direct colors. A definite improvement in color exhaustion and much cleaner and brighter shades are obtained. As an example, one dyer using 1 per cent T.S.P.P.* on the weight of goods estimated 25 per cent better color value when dyeing with a direct brown. Here, as well as in the case of sulfur colors, the T.S.P.P. is dissolved in the dye boil-up tank along with the dyestuff.

Sulfur Colors. Even in the dyeing of sulfur colors Tetra Sodium Pyro Phosphate has been found of use in yielding deeper and brighter shades of blacks and blues in single bath dyeings of raw stock or piece goods. With lighter colors a deviation in shade is introduced which must be compensated for. Typical dyeing formulae are as follows, using approximately 1/10 the weight of dyestuff as Tetra Sodium Pyro Phosphate:

TABLE III
Proportions of T.S.P.P. Used in Sulfur Dyeing

Dyestuff	8%	10%	15%
Conc. Sodium Sulfide.	5%	6%	9%
T.S.P.P.*	0.9%	1%	1.5%

Vat Colors. In washing vat colors a solution which formerly consisted of 1 per cent of an alkyl aryl sulfonate wetting agent and 2 per cent soda ash was changed to 1 per cent of the wetting agent and 1 per cent T.S.P.P.* (based on weight of goods) with very evident cleaner and brighter shades. The improved finish was judged more than equal to the slight extra cost from using the Tetra.

Cleaning Up Dye Machines. Where iron machines are used and are not in constant use they have a tendency to rust. This requires a cleaning of the machine before additional dyeings are run. Excellent results are obtained by using one (1) pound T.S.P.P.* and one-third (1/3) pound hydrosulfite of soda to each 100 gallons of water. This solution is boiled in the machine for ten minutes.

SILK

Due to its buffered alkalinity at the level of soap and its very excellent peptizing action on suspended solids, Tetra Sodium Pyro Phosphate is an adjunct of exceptional merit in degumming silk. It stops the formation of hard soaps which cause scum marks on the finished silk. Used in the proportion of 25-30 per cent based on weight of soap, Tetra Sodium Pyro Phosphate yields the following benefits:

a. Greatly improved degumming.
b. More uniform weighting due to the improved degumming.
c. No undegummed spots left on silk.
d. More uniform dyeing due to the improved degumming and weighting.
e. Lack of chafe marks, scum spots and streaks.
f. Lack of uneven dyeing caused by uneven weighting.
g. Elimination of rejects, adjustments in payments due to faulty finished silk, and added costs for refinishing.

A formula will have to be worked out for each particular plant. In order to give a basis for a start, the following formulae show the quantity of soap formerly used and the quantity of T.S.P.P.* and soap now being used with great success by an important silk weighting plant:

Using Soap Alone (Bath Used 4 Times)

	Fresh Bath	*Added for Each Additional Pass*				
		1	*2*	*3*	*Total*	*Per Pass*
	(*Pounds*)	(*Pounds*)			(*Pounds*)	(*Pounds*)
Soap ...	360	100	100	100	660	165

Using T.S.P.P. and Soap (Bath Used 5 Times)

	Fresh Bath	*Added for Each Additional Pass*					
		1	*2*	*3*	*4*	*Total*	*Per Pass*
	(*Pounds*)	(*Pounds*)				(*Pounds*)	(*Pounds*)
Soap	260	40	40	40	40	420	84
T.S.P.P.*.	50	20	20	20	20	130	26
	310	60	60	60	60	550	110

RAYON—CELANESE—NYLON

Tetra Sodium Pyro Phosphate will be found a useful addition agent to soap in the boil-off or scour of rayon. Celanese and nylon. Used in conjunction with a good olive oil soap, 2 pounds per 100 gallons of solution is a practical concentration, although it is preferable that proportions be based on the hardness of the water involved.

Because of the mildness of the alkalinity it is also useful in the dye formula where concentrations are usually reduced to 1 pound per 100 gallons or 1.2 grams per liter.

WOOL

Work in wool has not progressed to the degree that it has in other fields, and I have no comment to make on it at this time. There are some wool processes that have employed tetra sodium pyro phosphate successfully and

claim advantages in cost reduction in their operation. It has, however, not been generally used.

The author is indebted to A. B. Gerber, chief chemist of the Analytical Laboratory, C. B. Durgin and R. N. Footer of the Application Research Laboratory, and S. R. Clement of the Sales Department of the Phosphate Division of Monsanto Chemical Company and to the several textile mills from whom much of the data presented was obtained.

*T.S.P.P. is the abbreviation for Tetra Sodium Pyro Phosphate.

ONE HUNDRED AND THIRTIETH COUNCIL MEETING

THE Council held its 130th meeting at the Penn Athletic Club in Philadelphia on Friday morning, December 6, 1940. Present were President Carl Z. Draves, presiding; William R. Moorhouse, Treasurer; Louis A. Olney, Chairman of the Research Committee; William H. Cady, Elvin H. Killheffer, Robert E. Rose and P. J. Wood, Past Presidents; J. Robert Bonnar and Harold W. Stiegler representing New York; Charles A. Seibert representing Philadelphia; A. Henry Gaede representing Piedmont; Bertil A. Ryberg, Associate Research Director; and Harold C. Chapin, Secretary.

The Secretary's report of the 129th Council meeting and balance sheet of November 30, and the Treasurer's report of December 5, were accepted. A report from the Treasurer of the Rhode Island Section was approved. In accordance with the Tellers' report, Carl Z. Draves was declared President for 1941, and Roland E. Derby and Thomas R. Smith, Vice-Presidents. Albert E. Sampson was appointed to serve as Acting Treasurer during expected absence of Mr. Moorhosue, at times and with duties designated by Mr. Moorhouse, for period not exceeding six months. As recommended by the Piedmont Section, October 21 and November 1, 1941, in Pinehurst, N. C., were approved for time and place of the next General Meeting of the Association. A committee consisting of the Chairman of the Research Committee, the Treasurer and the Secretary, reported arrangements for insurance and annuity for Mr. Ryberg, which received approval of the Council.

Mr. Ryberg reported for the Committee on Publicity, Henry F. Herrmann, Chairman, outlining the contents and proposed circulation of the pamphlet presenting aims and accomplishments of the Association. A further appropriation was made for this purpose. It was recommended that all notices to the general press, save those regarding Sectional meetings, be submitted through Mr. Herrmann. Secretaries of Sections were requested to send advance notices of their meetings to the REPORTER, directly or through the Secretary of the Association.

Each of the following was elected to the class of membership specified, as of thirty days from publication of application, provided no objection be received meanwhile by the Secretary:

Senior

J. R. Adams	F. W. Fraim	J. N. Pantridge
J. E. Atwood	D. Fronmuller	A. E. Pinkney
F. F. Axelrad	J. H. Graham	H. E. Poore
J. J. Ayo, Jr.	W. F. Graney	W. D. Reid
B. Bailey	A. T. Grant	Q. M. Rhodes
S. L. Bass	C. W. Harris	E. K. Richman
W. Bowker	J. Hintermaier	D. S. Rosenthal
J. C. Broadmeadow	W. Horback	A. Rotcop
W. G. Chace	G. R. Howard	G. B. Shoemaker
W. E. Coakley	R. L. Hunter	J. W. Stewart
E. W. Clark	F. R. Jacobs	C. S. Tatum
F. J. Connell	S. Kende	P. G. Vlacos
R. H. Drukker	J. G. Kern	P. J. Witte
W. O. Fischer	O. F. Marks	A. S. Wittnebel
G. E. Fogle	J. H. Merscher	I. Wolder
R. N. Foster	H. L. Morgan	E. J. Young, Jr.

SECRETARY'S BALANCE SHEET—NOVEMBER 30, 1940

	Applications	*Dues, Regular and Reinstatement*	*Dues, Corporate and Sustaining*	*Miscellaneous*	*Bank Charges*	*Totals*
Brought forward, and received by Secretary, Aug. 1 to Oct. 12, 1940	$350.00	$63.50	$5.00	$247.41		$665.91
Received by Secretary. Oct. 12 to Nov. 30, 1940	470.00	6,993.00	1,780.00	854.87*	—$.75	10,097.12
Total received	820.00	7.056.50	1,785.00	1,102.28	— .75	10,763.03
Transmitted to Treasurer	820.00	6,916.50	1,775.00	1,102.28		10,613.78
Leaving to order of Secretary		140.00	10.00		— .75	149.25

Miscellaneous Items*

Annual Meeting Registrations (less $55, part of expenses)	$795.00	Reporters	.95
Dyed standards	7.00	Knitting tubing	17.00
Crock-meter cloth	9.92	Buttons	15.00
Year Books	10.00		$854.87

Junior

R. C. Bogle	K. R. Fox	F. A. Lyon
E. K. Camp	S. K. Freeman	G. R. Murphy, Jr.
B. R. Clanton	J. A. Harley	J. W. Place, Jr.
F. W. Corden	D. B. Keefer	J. J. Shedko
O. C. Elmer	R. K. Lyle	T. J. Scanlon
K. P. Ferguson	E. M. Lynch, Jr.	

Associate

M. C. Beard	M. J. Greene	T. P. Key
J. C. Cook	B. Gruenwald	W. D. Livingston
H. S. Drum	T. V. Hart	G. F. Ogan
H. Gowen	A. J. Hettesheimer	A. L. Sampson

Student

W. S. Bartenbach	R. J. Granger	A. A. Poitras
G. Brandt	C. F. Gregory	A. W. Powell
R. G. Buck	R. J. Guay	A. L. Roher
T. P. Caldwell	G. S. Higginbottom	G. R. Sedberry
W. P. Camp	W. A. Hoffman	S. F. Shafter
G. S. Cohen	S. L. Hornung	W. J. Sidebottom
J. E. DeKalb	M. L. Katz	E. P. F. Stewart
Y. C. Ding	J. R. Keim	S. M. Suchecki
L. C. Drye	J. L. Lee	A. C. Teichner
D. Echavarria	S. F. Lehman, Jr.	D. H. Thomas
R. A. Ewing	W. Lisien	W. R. Tyrie
G. W. Funderburk	S. M. Littlejohn	I. P. Wolf
E. A. Gleadall	W. Marcellino	G. E. Wood
	W. W. Platt	

The following were transferred from other classifications to Senior membership: J. A. Brittain, E. H. Driver, M. Einstein, J. A. Franklin, J. Klein, K. E. Leslie, W. A. Levins, G. L. Morrison. R. E. Ockford, J. H. Parechanian, E. W. Peters, F. L. Prescott. W. H. Shields. G. G. Slaughter, C. L. Simmons, E. G. Walker, J. H. Zahn, R. Zametkin.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary.*

ONE HUNDRED AND TWENTY-THIRD MEETING OF THE RESEARCH COMMITTEE

THE 123rd meeting of the Research Committee followed the Council meeting in Philadelphia on Friday afternoon, December 6, 1940. Present were Chairman Louis A. Olney presiding, W. D. Appel, K. H. Barnard, J. R. Bonnar, W. H. Cady, R. E. Derby, C. Z. Draves, A. Eavenson, E. Freedman, A. H. Gaede, M. Harris, E. H. Killheffer, D. P. Knowland, W. R. Moorhouse, G. A. Moran, D. H. Powers, R. E. Rose, B. A. Ryberg, C. H. A. Schmitt, C. A. Seibert, H. W. Stiegler, B. Verity, P. J. Wood, and H. C. Chapin, Secretary.

Mr. Cady presented a report from the Committee on Atmospheric Fading of Dyed Acetate Rayon, describing apparatus and procedure for testing susceptibility to gas fading through comparison with a dyed standard. This was voted a tentative standard method of the Association. Mr. Seibert, who had collaborated in preparation of the report, showed samples to illustrate a possible modification of method, involving a final warm pressing to emphasize color change.

Following extended discussion of rumors of dermatitis from textile products, particularly through finishing materials, Messrs. Barnard, Freedman, Killheffer and Powers were appointed by the Chairman a committee to suggest methods of systematic investigation.

There was discussion as to how the Association system, giving the fastest dyeing the highest number, might be reconciled with common practice of rating best materials first. The Association system was defended as leaving still higher numbers available for the better products of the future. Because this has already been adopted generally by dyestuff companies, textile manufacturers, and departments of the Federal Government, hope was expressed that if the retail trade requires another system, it may employ descriptive terms other than numbers.

Need for immediate revision of the fastness to chlorine test on cotton, toward more definite specification of hypochlorite and alkali content, was again stressed by Mr. Seibert. With reference to this and other tests for which dyed standards have been prepared by the Association, it was the consensus of opinion that such standards are integral parts of the tests, which should always appear in printed statements thereof, even though dyeing details be omitted.

There was consideration of what tentative test methods might be made official, and tests S-2, S-3, S-4 and S-5 for fastness of dyed or printed silk to fulling, degumming, stoving and peroxide bleaching, were so reclassified. In reply to the suggestion that the test for fastness of dyed wool to carbonizing might be made more specific, Mr. Derby said that it would be; but further stated that use of the laboratory test in question is confined to dyestuff dealers, and to textile manufacturers for whom a test in the carbonizer itself is more customary. It was voted that the carbonizer test be retained as recommended procedure. With regard to perspiration fastness tests, possibility of making the oven method official was discussed, also the need of further work on standards of lower fastness rating.

With reference to final pressing of cotton in washing tests, attention was called to greater change of staining white cotton between iron and colored sample in certain tests recognized by the Association as equivalent to its own tests; though in its own the sample is placed next the iron, with white cotton below. It was reported that new Launderometer balls are available, free from the tendency to stain wool at higher temperatures which balls weighted with lead compounds have shown. In shrinkage requirements, on rayon, the trade was said to desire a small amount of shrinkage in washing to compensate for subsequent stretch in ironing.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary.*

MEMBERSHIP APPLICATIONS

Senior

Catherine B. Armstrong—Textile Technologist, General Dyestuff Corp., New York, N. Y. Sponsors: C. Z. Draves, H. C. Borghetty.

Alfred Griffin Ashcroft—Production Engineer in Charge of Research and Control, Alexander Smith & Sons Carpet Co., Yonkers, N. Y. Sponsors: W. v. Bergen, J. R. Bonnar.

Morton H. Baker—In Charge Thread Finishing Research, American Thread Co., Willimantic, Conn. Sponsors: A. Veevers, A. C. Persons.

Patrick Thomas Baxter—Superintendent, Atlanta Woolen Mills, Atlanta, Ga. Sponsors: H. G. Smith, W. C. Ragin, Jr.

G. H. Boyd—Salesman, E. I. du Pont de Nemours & Co., Inc., Chattanooga, Tenn. Sponsors: D. S. Rion, M. W. Moore.

Clifford Bradley—General Manager, Farwell Bleachery, Lawrence, Mass. Sponsors: M. J. Lane, W. E. Durgin.

Basil D. Browder—Asst. to President, Riverside & Dan River Cotton Mills, Danville, Va. Sponsors: R. D. Howerton, D. S. Quern.

John Franklin Byrd—Textile Chemist, Marshall Field Mfg. Div., Spray, N. C. Sponsors: J. B. Wray, R. H. Tuttle.

Mario D. Casapulai—Textile Colorist, United Piece Dye Works, Paterson, N. J. Sponsors: B. A. Roberts, J. E. Meili.

Craven Hulton Casper — Head Dyer, Amazon Cotton Mills, Thomasville, N. C. Sponsors: T. R. Smith, D. S. Quern.

Morris Lewis Chandross—Director of Testing Laboratory, Abraham & Straus, Inc., Brooklyn, N. Y. Sponsors: L. Kapelsohn, R. J. Singer.

William Gutig—Chemical Engineer, Atlantic Dye Works, Paterson, N. J. Sponsors: J. E. Meili, O. H. Genssler.

William J. Harrison—Chief Chemist, The Trion Co., Trion, Ga. Sponsors: D. A. Torrence, Jr., W. E. Hadley.

Baptiste J. Lanza—President, Lanza Silk Dyeing Co., Paterson, N. J. Sponsors: L. J. Roos, A. Booth.

Paul Logue — Sales Development, Monsanto Chemical Co., St. Louis, Mo. Sponsors: J. C. Harris, J. L. Crist.

William Francis Macia—Rayon and Staple Fiber Technologist, The A. M. Tenney Associates, Inc., New York, N. Y. Sponsors: J. E. Meili, H. DeW. Smith.

Thomas F. Malloy—Dyer, Botany Worsted Mills, Passaic, N. J. Sponsors: P. M. Otto, H. Ehrhardt.

A. Bingham Owens—Asst. Export Sales Mgr., Organic Chem. Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del. Sponsors: C. A. Seiber, G. H. Schuler.

Angelo J. Ramonda—Superintendent of Dyeing, Farwell Bleachery, Lawrence, Mass. Sponsors: M. J. Lane, W. E. Durgin.

William E. Reid—Superintendent of Finishing, The Trion Co., Trion, Ga. Sponsors: A. H. Gaede, M. W. Moore.

Harry Richardson—Asst. Dyer, Ayer Mill, American Woolen Co., Lawrence, Mass. Sponsors: H. E. Wieland, E. A. Buthmann.

Edward Schlaginhafen—Dyer, Durham Dye Works, Weehawken, N. J. Sponsors: J. H. Ciciva, A. F. Johann.

Mitchell A. Sieminski — Textile Technologist, Atlantic Research Associates, Inc., Newtonville, Mass. Sponsors: G. H. Hotte, H. H. Broadfoot.

William R. Tornquist—Superintendent, Clearwater Manufacturing Co., Clearwater, S. C. Sponsors: W. B. DePass, C. M. Robbins.

Anton Theodoor von Huben—Technical Manager, Van Houten Steffan en Co., Padang, Sumatra, Netherlands East Indies. Sponsors: C. A. Seibert, G. H. Schuler.

George G. Wellworth—Mill Manager, W. Lowenthal, Inc., Rossville, Ga. Sponsors: J. Anderson, F. F. Myers.

Kurt Wenzel—Chemist, E. I. du Pont de Nemours & Co., Inc., Shanghai, China. Sponsors: C. A. Seibert, G. H. Schuler.

Alva Taylor Wilson—Chief Chemist, Whittemore-Wright Co., Inc., Charlestown, Mass. Sponsors: J. E. Travers, E. S. Chapin.

Lester Jerome Workman—Salesman and Demonstrator, L. Sonneborn Sons, Inc., New York City. Sponsors: M. N. Manfra, A. Moscowitz.

William R. Zimmerle—Dyer, Swiss Bleaching & Lace Finishing Co., Weehawken, N. J. Sponsors: J. H. Ciciva, A. F. Johann.

Junior

Joseph J. Black—Dyer's Asst., A. M. Townson Co., Philadelphia, Pa. Sponsors: A. M. Townson, J. E. Goodavage.

Arthur S. Davis—Textile Chemist, United States Testing Co., Hoboken, N. J. Sponsors: J. H. Skinkle, H. C. Chapin.

Julian C. Freel—Asst. Dyer, Mandeville Mills, Carrollton, Ga. Sponsors: H. G. Smith, J. J. Haggerty.

William G. T. Mepham—Textile Chemist, Toronto Carpet Mfg. Co., Toronto, Ontario, Canada. Sponsors: L. P. Brick, C. D. Ehrengart.

Robert W. Nock—Textile Colorist, E. I. du Pont de Nemours & Co., Inc., Technical Laboratory, Wilmington, Del. Sponsors: R. A. Shimp, G. W. Grant.

Fred R. Sheldon—Sales and Service, Buffalo Electro-Chemical Co., Philadelphia, Pa. Sponsors: J. F. McCoy, J. P. Conaway.

James E. Turner, Jr.—Chemist, Wiscassett Dye Plant, Albemarle, N. C. Sponsors: T. R. Smith, D. S. Quern.

Associate

Harlan C. Brown—Librarian, The D. H. Hill Library, North Carolina State College, Raleigh, N. C.

John W. Haught—Division Head, Chemical Research Dept., E. I. du Pont de Nemours & Co., Inc., Arlington, N. J.

Charles S. Humphrey—Salesman, Onyx Oil & Chemical Co., Ltd., Toronto, Ontario, Canada. Sponsors: L. P. Brick, C. D. Ehrengart.

Clarence A. Ross—Gastonia Combed Yarn Corp., Gastonia, N. C.

Richard Seiferheld—Manager Production Dept., N. Erlanger Blumgart & Co., Inc., New York, N. Y.

Frederick R. Thus—President, Reading Dyeing Co., Inc., Reading, Pa.

Student

Clarence W. Bachman — Philadelphia Textile School. Sponsor: E. C. Bertolet.

William J. Sidebottom—Lowell Textile Institute. Sponsor: L. A. Olney.

Arthur C. Teichner—Lowell Textile Institute. Sponsor: L. A. Olney.

Donald H. Thomas—Lowell Textile Institute. Sponsor: L. A. Olney.

Wallace R. Tyrie—Lowell Textile Institute. Sponsor: L. A. Olney.

Irving P. Wolf—Lowell Textile Institute. Sponsor: L. A. Olney.

Raymond J. Guay — Bradford Durfee Textile School. Sponsor: L. B. Coombs.

Transfer Applications: W. A. Haines, C. G. Stover, J. T. Bohannon, G. D. Marrocco, R. E. Noyes, R. J. DeWitte, H. S. Schenker, P. G. Wear.

MEETING, MID-WEST SECTION

THE meeting of the Mid-West Section was held at the Bismarck Hotel, Chicago, Illinois, on Saturday, December 7, 1940.

The afternoon meeting was called to order at 3:30 P. M. by Chairman Herman Boxser, who introduced Miss Laura E. Pratt of Sears, Roebuck & Company. An illustrated talk was presented on "Test Methods for Consumer Appeal." This talk was well received by about forty-five members and guests.

Dinner was served at 7:00 P. M. and attended by 62 members and guests.

The business meeting was called to order at 8:00 P. M. and the reports of the Treasurer and Secretary were read and approved.

The chairman of the Nominating Committee, Elmer Smith, and the other members, Frederick Wilhelm, Edward Siegrist and Robert Griswold, announced that the following were selected for the coming year:

Chairman, Archie Alexander; *Vice-Chairman*, Frederick Wilhelm; *Treasurer*, James Morrison; *Secretary*, J. G. Stott; *Representative to the National Council*, Virgil Hartquist; and *Sectional Committee*: L. B. MacFarland, Elliot Morrill, Dr. Harold Tucker and Robert Griswold.

Herman Boxser then made his farewell address and turned over the meeting to Archie Alexander who made his opening speech. James Morrison made a motion, which was seconded and approved, that an award of a cash prize be given for the best paper presented by a member at an afternoon meeting. The business meeting was then adjourned.

Archie Alexander then introduced H. Millson of the Calco Company. Mr. Millson gave a talk on "Microscopic Observation of Wool Dyeing" with slides to illustrate. This was the second section of his experiments in this line. The talk was well received and some discussion followed.

The meeting was then adjourned at 11:00 P. M.

Respectfully submitted,

J. G. STOTT, *Secretary*.

Twenty-first

Annual Meeting

and Convention

October 31-November 1, 1941

Carolina Hotel, Pinehurst, N. C.

TRADE NOTES • NEW PRODUCTS

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson Street, New York, N. Y., announces release of circulars describing the following products:—

Sulphon Yellow RS Extra—an acid yellow producing fairly bright shades. The dyeings are said to possess fairly good to good fastness to light, good fastness to washing and to perspiration, and good to very good fastness to fresh and sea water. It is recommended by the manufacturers primarily for the dyeing of knitting yarns for sweaters and bathing suits, and also of loose wool for blankets. It is stated that, due to its good fastness to fulling and its good resistance to chrome, it is useful as a shading color in chrome dyeing. Possessing good affinity from a neutral bath, it is well suited also for the dyeing of unions of cotton or rayon and wool. It may further be used for the dyeing of both pure and tin weighted silk. The product can be supplied in a quality suitable for goods which are rubberized after dyeing. Circular G-213.

Soromine FW Paste—a softening and finishing agent well suited for use on rayon, rayon mixtures and cotton. It is a white paste of neutral reaction, readily soluble in water. It is not cation-active and can, therefore, be applied directly from the dyebath. It is said to impart a soft, smooth handle, which is not affected by cold rinsing. The product cannot, however, be used in the same bath with basic dyestuffs. It can be applied in conjunction with soap, Turkey Red Oil preparations and most other finishing agents to produce any degree of softness or finish desired. Circular G-212.

Benzo Bordeaux 7BA—a direct dyestuff producing fairly bright shades of a bluish red on the various forms of cotton and rayon. The dyeings can be discharged to a good white with Rongalite C. It is well suited also for the dyeing of unions consisting of cotton and wool. It is stated that only a small amount of a shading color need be added to produce completely uniform effects. Circular G-214.

Monochrome Red FGA Conc.—a chrome color which may be applied by the afterchrome or the monochrome method, and can also be dyed on a chrome mordant. It is said to produce shades of a very good-to-excellent fastness to light and of very good fastness to washing, to fulling, to water, to salt water, and to perspiration. It is stated that it is particularly useful where fastness to light and to fulling is required on loose wool, slubbing, piece goods, and yarns. The product is level dyeing. Circular G-215.

• FEATURED IN "LIFE"

The photo of the girl model used in the advertisement of Onyx Oil & Chemical Co., Jersey City, N. J., which appeared in the January 6th, 1941, issue of the REPORTER, was featured in "Life" magazine in an article appearing in the issue of December 16th, 1940. The article, which was profusely illustrated, told just how a studio goes about the matter of preparing and taking a hosiery photograph. The many obstacles and handicaps encountered in taking a fine photograph and the patience and skill required in overcoming them were discussed. The photo used in the Onyx advertisement, which is reproduced above, was posed and taken by the Black Box Studios of New York City and was presented by "Life" as an outstanding example of fine art in modern hosiery photography. The model was Miss Evelyn Carmel.

Katigen Olive GG-CF—a sulfur color said to be of good fastness to washing and of very good fastness to light. It is said to be valuable both as a self-shade and for combinations with other sulfur colors. It is stated that it possesses good solubility and good levelling properties and is well adapted for use in the jig and in pressure machines. It is suitable for dyeing goods which are to be rubberized. Circular G-216.

• REPRINT ON COTTON FIBERS

Reprints of an article entitled "Cotton Fibers: Constitution, Structure and Mechanical Properties" by Dr. R. F. Nickerson are available to readers of the REPORTER from the Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh, Pa. Dr. Nickerson is a member of the research staff of the Institute. The article is reprinted from *Industrial & Engineering Chemistry*, November, 1940. An abstract describes the contents of the article as follows:

"Data on the constitution of raw cotton are presented and discussed. Various theories of fiber structure are reviewed. A fiber structure in which the component fibrils are formed from many unit crystalline areas linked by primary-valence glucose chains appears to be the most acceptable. The crystalline units probably represent the crystallization of portions of adjacent glucose chains.

"Such mechanical properties as tensile strength, elasticity, plasticity, swelling and elastic aftereffect are summarized. A structure of the type just mentioned is compatible with the properties.

"One object of the review has been to assemble for ready reference the available data on cotton fiber constituents and properties. These data are presented in their relation to fiber structure wherever it has been possible."

• NEW RAYON FIBER

A new rayon fiber, said to possess a high degree of permanent crimp, has been developed by the Rayon Department of E. I. du Pont de Nemours & Company, Inc. Lacking as yet a more formal title, it is known by its laboratory designation, "Fiber D." Tests indicate that fabrics containing Fiber D possess certain characteristics now available only in wool.

The new fiber has performed satisfactorily in rugs, carpets, upholstery materials and wall coverings. It can also be used in other decorative fabrics, including draperies, automobile upholstery and plushes.

It is stated that the crimp, which gives bulk and loft to the yarns and the wool-like appearance and feel of the fabrics, is an inherent characteristic of Fiber D. If it is partially removed during processing, it may be recovered by simple treatments. This is a fundamental difference between Fiber D and earlier crimped rayon from which the crimp is easily lost.

An outstanding characteristic of the fiber is a smooth cross-section, as contrasted to the "crenulated" or ridged pattern of standard rayons. This is claimed to enable fabrics made of it to shed dirt more effectively.

As with other rayons, Fiber D may be dyed to brilliant clear colors even in the lightest shades. This is of particular interest to designers of rugs, who have been unable to use light colors unless working with the carefully selected and expensive white wools. Luster can be controlled, as in other rayons. The fiber is said to be mothproof and to take readily to treatment with anti-mildewing and fire retardant preparations.

Carpet and rug manufacturers have displayed particular interest in the new fiber, as a safeguard against unstable prices and uncertainty of supply in carpet wools, many grades of which are imported from India, China, Persia, Iraq, Syria, and other remote areas. It is stated that abrasion tests measuring the wearing properties of carpets showed "substantial improvement" over carpet wool in this service. Covering power of the fiber was described as extraordinarily high, giving a consequent richness to the fabric. When treated with "Zelan" textile finish, Fiber D acquires a soft hand that is said to be indistinguishable from carpet wool.

In lighter yarns Fiber D is said to give to fabrics the feel and appearance of worsted. One manufacturer is offering a varied line of high grade upholstery and drapery fabrics. One of these, a wall covering fabric has just been installed in the new National Gallery of Art in Washington as a background for the Mellon art collection. The material, woven to give a hand-loomed effect, has a strength permitting it to be drawn tight without buckling. It was flame-proofed and also treated against silverfish, tiny pests frequently found in plaster and masonry.

As is true of wool, Fiber D has been used largely, but not solely, in infrequently washed fabrics. Its rounded smooth cross-section answers the specifications of patents owned by the Denicron Corporation and it is consequently being used in fabrics bearing the "Denicron" label. Du Pont officials emphasized that the company has no connection with the Denicron firm, and that Fiber D's suitability under current Denicron patents is coincidental.

● CHANGE OF NAME

Hatch Textile Research announce that they have succeeded to the laboratory and testing business of Hatch Textile Research, Inc. (a corporation) as of Jan. 1, 1941. There will be no change in management or policy.

The year 1941 marks the 15th anniversary of this testing company which started business in 1926 with only one client, a prominent department store. Since then the Hatch firm has served the New York textile trade and retail stores in meeting their testing requirements. Herbert Harriman Hatch, known in the textile market locally since 1909, remains director of the testing laboratory.

● SPEAKS TO STUDENTS

Frank D. Levering, President of Eavenson & Levering, Camden, N. J., addressed the students and staff of the Philadelphia Textile School on Thursday atfernoon, January 9. His topic was "The Place of Commission Scouring and Carbonizing in the Wool Textile Industry." Mr. Levering is a former graduate of the School, secretary of the Philadelphia Wool and Textile Association, and a vice-president of the National Association of Wool Manufacturers.

● USE OF ZELAN

Use of textiles treated with "Zelan" durable water repellent increased more than ten fold during 1940, officials of E. I. du Pont de Nemours & Company announced recently.

Cotton sport jackets, rainwear, and hunting clothes, among the first garments treated with "Zelan," still consume a major portion of the yearly production. It is also announced that the United States Army is now using treated fabrics for winter jackets. The jackets are wool lined and modeled similarly to the ordinary ski blouse.

Among the new uses for the treated fabric are children's play clothes, boys' corduroy suits, and shirts. Others include hosiery, heating pads, galoshes, luggage, evening wraps, shower curtains, mattresses, hats, handbags and pillows.

One hundred and fifty children's garments were submitted for tests to home economics groups in universities and various women's organizations, throughout the country. It is stated that the tests by these groups revealed that the treated materials were "most satisfactory" for children's clothing. Mud can be brushed from the treated fabric, while untreated clothing must be washed or dry-cleaned.

The employment of "Zelan" on sail cloth is increasing. Results of tests conducted last year with treated sails have created an active demand. It is said that addition of the finish does not stiffen or bulk the fabric.

Yachting costumes of wind-resistant poplins, corduroys and spun rayons, to protect against spray, rain and fog are becoming an important outlet for the repellent.

● CIBA REVIEW

The subject matter of *Ciba Review*, Number 36, for November, 1940, concerns itself with "Indian Costumes." Contents are described by the following titles: Periods of Civilization and the Development of Dress in India; Costumes of the Peoples of India; Indian Textiles, Their Manufacture, and Their Patterns in the 19th Century and at the Present Day; Notes; Scientific and Technical Notes.

● CLARION PURCHASES PLANT

Clarion Chemical Corporation has recently purchased a large plant on the corner of Main and George Sts., Newark, N. J. Modern equipment has been installed for the production of its various textile products. Dr. H. E. Smith continues as executive vice-president and technical director.

● EASTMAN ENTERS DYE FIELD

Announcing its entry into the acetate dye manufacturing field, the Tennessee Eastman Corporation is offering as one of its first products a new blue dye said to be of exceptional gas fastness for acetate rayon fabrics.

It is claimed that the new dye provides a complete range of blue tones, from the deepest shades to light pastel tints, with a freedom from gas or atmospheric fading, heretofore unobtainable except possibly over a limited range of blue tones. Another unique advantage of the dye, which is called Eastone Blue BB, is said to be that it shows no tendency to turn pink or fade off-hue under severe light conditions.

Dyers are already employing the dye on acetate rayon lining fabrics. It has also been tested on many types of outerwear fabrics, and is applicable to taffetas, satins, and sharkskins in the all acetate group, as well as to crepe mixtures and Teca Blends, for either solid or cross-dyed effects. It is stated that tests by independent testing laboratories indicate that the dye has exceptional color fastness to washing, dry cleaning, and ironing, and that it is also fast to crocking or discoloring from perspiration.

The use of Eastone Blue BB will not be confined to fabrics containing Eastman acetate yarns. It is stated that dyers will be permitted to apply it to fabrics containing any kind of acetate rayon, in order that its gas-fast qualities may be made available at once to the entire industry.

Simultaneously with the introduction of Eastone Blue BB, Tennessee Eastman offers a complete line of similar dyes in the Eastone series covering the rest of the color range, and available for use on any fabric containing acetate rayon.

As one of the foremost producers of cellulose acetate, Tennessee Eastman has realized the opportunities for specialized research on dyes for that material. In setting up its present research and production facilities they state that they hope to render an important service to the textile and plastics fields in which cellulose acetate is so widely used.

● PURCHASE SORBTEX LICENSE

Century Ribbon Mills, Inc., New York, has purchased an exclusive license to use the Sorbtex Process on typewriter ribbons. The license covers application of the Sorbtex Process to all types of typewriter ribbons, including cotton, silk, linen, rayon, nylon, and also blended fiber typewriter ribbons, and includes woven edge, fused edge and cut edge ribbons.

The effects of the Sorbtex Process on typewriter ribbons are said to be the securing of uniformity in the appearance of the typed page and greatly increased durability and much longer life for the ribbon.

• TECHNICAL NOTES • FROM FOREIGN SOURCES

A Machine for Testing the Fastness to Rubbing of Dyed Materials

Derrett-Smith and Bradley—J. Soc. Dyers & Col., 56-261, *June*, 1940.—In a communication from the Linen Industries Research Association of Ireland there is given a detailed description of a machine designed to test the fastness to rubbing of dyed yarns and fabrics. This "rubbing machine" differs in several details from the standard Crock Meter of the A.A.T.C.C. (1939 Year Book, page 197), and a brief comparison of the two may be of interest.

The Linen Research Association instrument is a decidedly more elaborate mechanism, motor driven, and is designed to rub the dyed material upon white cloth, which is the reverse of the Crock Meter operation. Instead of employing a round disk or finger for the rubbing, the test material is fastened around a one-inch hard wood block which has a rounded bottom, permitting only about ⅛-¼ inch length of the colored goods to come into actual contact with the white cloth. During the test operation the block is fixed in a light metal frame, the weight of both block and frame together amounted to 210 grams.

Over a raised aluminum platform is stretched a two inch wide strip of white cloth, the inner end clamped fast and the outer end, which hangs down from the platform edge, kept taut by means of an attached weight. The dyed material is fastened in place around the wood block, which is then placed in its metal frame, pressing the colored material against the white strip with a weight of 210 grams. A geared motor drives a heart-shaped cam from which connecting rods are fastened to the frame of the rubbing block and impart to it a reciprocating motion of practically constant speed in its travel of approximately 5½ inches back and forth over the white cloth. The motor is geared to a speed of 42 r.p.m., thus giving 84 rubs per minute, a rate which requires an operating time of only ½ to 2½ minutes for tests based on 50 to 200 rubs. A mechanical counter attached to the motor records the number of rubs made on each test. The white fabric may be dampened if it is desired to test fastness to wet crocking.

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Silk Fibroin—

(Concluded from page 28)

base would be bound at higher pH values at which, however, the estimation of base bound and of pH would be attended by such large errors as to make the experimental data unreliable.

Additional evidence for the conclusion that the phenolic hydroxyl groups of tyrosine bind base is provided by the data on the titration of methylated fibroin with potassium hydroxide, given in Table IV. In this material all but approximately one-fifth of the tyrosine had been methylated. The titration curve (solid circles, Figure 1), is shifted toward higher pH. Up to pH 9.5 very little base is bound and at any given pH the amount bound is very much less than in the case of the unmethylated fibroin. This greatly decreased base-binding capacity of the methylated fibroin lends support to the interpretation of the very steep portion of the curves for non-methylated fibroin as being due to the titration of the phenolic hydroxyl groups of tyrosine.

TABLE IV
Amounts of Base Bound by Methylated Fibroin at 0° C.

pH	*Base Bound M—eq./g.*
8.74	0.005
9.48	.005
12.15	.037
12.55	.061
12.84	.091
13.47	.166
14.14	.283

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Cotton Fibers—

(Continued from page 30)

resultant change in the mobility curve. Numerous instances exist in which a physical change, such as alteration of the degree of orientation, has been shown to change the electrokinetic properties of substances. Kanamaru, for example, has observed that the zeta potentials are different for rayons spun of the same material, but of varying degrees of orientation[15], and a similar observation has been made on nylon by Sookne and Harris[14]. Harrison found a large decrease in the (negative) zeta potential of cotton after mercerization[16], and since the small changes observed in the present investigation are in the same direction, they could possibly be caused by alteration of the physical character of the fiber surface.

It will be noted that the depectinized samples of cotton exhibit reversal of charge below about pH 2.5 and accordingly are isoelectric at that pH. However, it has been reported[17] that cellulose has no true isoelectric point and that reversal of the charge in the more acid solutions resulted from hydrolysis at the surface of the fiber. Although these conclusions arose from investigations on wood

cellulose, it nevertheless appeared advisable to determine whether any irreversible effects had been produced under the conditions of the present studies. For this purpose, the following experiments were performed. 1.5-g. samples of ground, depectinized (8 hour) cotton were soaked in 400-ml. portions of hydrochloric acid-potassium chloride solutions (0.02 *M* ionic strength) having pH values of 1.7, 2.5, and 3.3. These solutions were identical with those used in obtaining the data shown in Fig. 1. After 1 hour, the samples were filtered off on a sintered glass funnel and thoroughly washed with running distilled water. Water suspensions of each of the three samples as well as of a sample of the untreated depectinized cotton were then prepared and sufficient acetic acid and sodium acetate added to bring the pH value of each solution to 5.6 and the ionic strength to 0.005 *M*. The samples were allowed to remain in contact with the buffer solutions for 24 hours, after which the mobilities of the particles were measured. No significant differences between the acid-treated and untreated samples were found and therefore it was concluded that no irreversible changes in the surface potential of cotton were produced by the dilute solutions of acid employed in the present work.

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• BOOK • REVIEWS

Handbook for Chemical Patents. *Edward Thomas, A.B. 270 pages. Price* $4.00. *Chemical Publishing Co., Inc., New York City, N. Y.* 1940.

The author, a leading chemical patent attorney, and one time assistant examiner in the U. S. Patent Office, presents in a clear and interesting style the subject of chemical patents, and chemical patent litigation. While only about fifteen per cent of all patents are chemical patents, the fact that more than fifty per cent of all patent litigation concerns chemical inventions, is indicative of the importance of this branch of patent law. The author believes this is due to the difficulties inherent in chemical inventions, which result in poorly prepared patents. The author hopes that the suggestions he has given for the writing of specifications for such patents may overcome much of the difficulty.

The following headings of chapters will indicate the scope and completeness of this book:

I. You Can't Keep a Chemical Secret.
II. Patents Raise Problems.
III. The Nature of a Patent.
IV. Suggestions for Writing the Specifications.
V. Inventions Under the Patent Statute; Their Nature and Their Date.
VI. Anticipation of a Process.
VII. Anticipation of a Composition of Matter.
VIII. Anticipation of a Manufacture.
IX. Anticipation of a Chemical Apparatus.
X. Anticipation of Prior Publications.
XI. Suggestions for Writing the Claims of a Patent.
XII. Interpreting the Specification.
XIII. Infringement of a Process Claim.
XIV. Infringement of a Composition of Matter Claim.
XV. Infringement of a Manufacture Claim.
XVI. The Patent Application in the Patent Office.
XVII. Amending the Patent After It Issues.
XVIII. Double Patenting and Division of Application.
XIX. Interferences.
XX. Assignments and Licenses and Employees.
XXI. Patent Suits.
XXII. Patent Evidence.
XXIII. Foreign Patents.

The Tables of Cases; and Citations of Law given in the last fifty pages as well as the very complete index will be a great help to the student of Patent Law.

Index to A.S.T.M. Standards, 1940-1941. The Index and gives information on all of the 952 standards as of Degives information on all of the 952 standards as of December 1, 1940. The Index will be of service to anyone wishing to ascertain whether the Society has issued standard specifications, test methods, or definitions covering a particular engineering material or subject and it will also be of help in locating the standards in the volumes where they appear.

All items are listed in the Index under appropriate key-words according to the particular subjects they cover and as a further convenience a list is given of the specifications and test in numerical sequence of their serial designations.

Copies of this 172-page publication may be secured without charge upon written request to A.S.T.M. Headquarters, 260 S. Broad St., Philadelphia. Pa.

Soap Manufacture. *J. H. Wigner. Ph.D.* 168 *pages. Chemical Publishing Co., Inc.,* 148 *N. Lafayette Street, New York, N. Y. Price* $4.00. *A handbook for students and those engaged in the Manufacture of Hard Soap.*

The object of this book is to provide information of a practical kind for the use of those engaged in the day to day tasks of soap works, and the matter treated of has been mainly confined to the processes of soap boiling proper. Practical soap makers are liable at any time to be confronted with new problems. It is of little assistance when dealing with these to have information limited to the mere description of the processes under normal conditions. In this book much information is given as to the real nature of the processes, and the reader is given an insight into the course of the operations which should enable him to decide for himself on the best course to pursue when changes in the conditions are unavoidable.

The following headings of the nine chapters of the book will give a general idea of its comprehensive contents:

I. Introduction—the conventional method of soap boiling—properties required in commercial soaps—soaps for various technical purposes.
II. Raw Materials.
III. Experimental Boils.
IV. The Soap-Boiling Process.
V. The Soap-Boiling Process (continued).
VI. Commercial Soaps.
VII. Glycerin in the Spent Lyes.
VIII. Enrichment of Lyes Weak in Glycerin.
IX. Production of Crude Glycerin from Spent Lyes.

The Merck Index. *Fifth Edition.* 1,060 *pages. Merck and Co., Inc., Rahway, N. J. Price* $3.00. 1940.

An encyclopedia for the Chemist, Pharmacist, Physician, Dentist and Veterinarian containing useful scientific data and other information on the physical, chemical and medicinal properties, as well as the various uses, of chemicals and drugs.

The Merck Index is so well known to most chemists that it needs but little introduction. The first edition was published in 1889 and since that time the work has been extended through subsequent editions so that at the present time it represents an extensive and authoritative reference book. Practically every compound of technical or scientific interest is described, which is indicated by the fact that specifications are given for 5,900 such products. In the section on coal-tar colors used in foods, drugs and cosmetics, one hundred and thirteen are described. A detailed list with specifications is given for many indicators used in testing both qualitative and quantitative with full directions for their preparation. The latter part of the book covers 4,510 chemical, clinico-chemical reactions, tests and reagents all described under the author's name. Details for the preparation of culture media, fixatives and staining solutions is also given and various tables of weights and measures.

The Merck Index fills a place in the chemists' library which is scarcely duplicated by any other book, at least of its size and cost.

All About Wool. *Diccionario De Terminos Laneros. Paul Link.* 150 *pages. Price* $2.00. *Published by G. E. Stechert & Co., New York.* 1940.

A few years ago the author began to compile brief definitions of technical terms and subject matter relating to sheep and wool. He made this compilation in both Spanish and English with a view of later offering to his Spanish speaking students a book that would aid them and enable them in grasping the proper meaning of the technical terms concerning their special fields of studies. It was the original intention to publish the book only in Spanish but as the work progressed it was realized that its value would be greatly enhanced by publishing it also in English for the benefit of those who could not read Spanish.

The book is unique in that the entire work is published in a two column arrangement where the Spanish is in one and directly opposite is the English translation. The author realizes that the first edition of a book of this kind can hardly be complete, but it is his intention to continue the compilation so that future editions, if published, may be more complete. In spite of the author's modesty, we should say that the first edition was remarkably complete as far as terms included are concerned and it embraces a wide range of subjects—heredity, breeding, common diseases, and breeds of sheep, also terms used in the science, commerce, manufacturing and marketing of wool.

• OPEN • FORUM

53—We should be pleased to have information concerning the block printing of yarn. Also if anyone produces special machinery for this purpose and if yarn is printed to any extent by this method. We should also like to know how extensively block printing in general is used in the United States and if there are concerns who make a business of manufacturing the necessary blocks.—**C. C. E.**

Answer—"Block printing" of yarn as we know it in the literal sense of the term, is to my knowledge no longer generally employed in the United States. The reason for its decline was the clumsiness of the blacks, the many handlings to which the yarn was necessarily subjected and the fact that, through this handling, the condition of the finished yarn was unsatisfactory to the trade.

Printed yarn requires a pasted dyestuff which should have the fastness to withstand a steaming or ageing to "set" the shade, a washing out of the gum, and should withstand either a severe extracting to prevent "lay-marks" or "marking-off" or a protracted hanging in the dry room or conditioning room. Furthermore, even penetration of the skein proper is difficult to achieve, and a proper set-up to prevent the yarn from

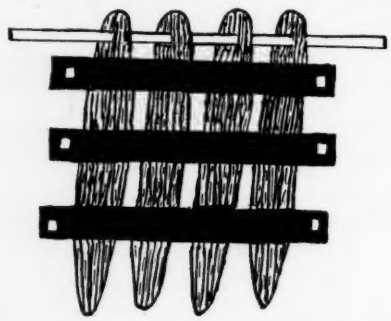
slipping during printing is not easy to make; in fact, it is practically impossible.

Off hand, I don't know of any commercial manufacturer of blocks in the business at the present time, although they are easy to make either of wood, linoleum or metal. Nevertheless there is a demand for the process and a quality job would probably increase the demand.

Nearly all skein dyers know of the so-called knot and clamp dyeing processes. Following are two very ingenious methods which can be used for any fiber and with probably any class of dyestuff.

Clamp Dyeing

Heavy rubber strips secured by lugs at each end are used as shown in the illustration. The intervening



spaces are dyed in the usual manner for the dyestuffs used. As many repeats as desired or are practical will give multicolor effects. Care must be taken not to use too hot a bath when dyeing acetate rayon as the yarn has a tendency to fuse under pressure of the rubber at high temperatures. The rubber strips can be any desired width. They are usually reinforced with a light metal such as aluminum. The lugs should be strong enough to necessitate tightening with a wrench.

Machine Dyeing

This, I believe, is a patented process, and I can give only a general idea. An intricate machine composed of a number of layers (or shelves), removable frames which form compartments when laid on the shelves, suitable pumps, valves and dyestuff reservoirs are necessary. Perforated hollow needles penetrate each compartment vertically.

Let us assume that the machine has five shelves. The required number of skeins is laid on the first shelf. A frame, so constructed as to form water tight compartments, is placed and secured over the yarn. A needle runs through each compartment being used, and penetrates the five shelves. This is repeated on the remaining shelves. The dye liquors are forced through each needle, and results in a very fine printed effect. Of course, the description of the machine is very sketchy and probably very inaccurate, but I am told that this process yields effects as small as one-quarter inch wide, and that the condition of the yarn remains unaltered.—**A. B.**

• CLASSIFIED • ADVERTISEMENTS

WANTED: TEXTILE RESEARCH ASSISTANT: Recent textile school graduate, some experience cotton dyeing or allied research. Age 21-25. Excellent opportunity advancement established medium-sized company, highest rating, out of town. State full details first letter including experience, age, education, religion, approximate salary. Send recent snapshot not to be returned. Write Box No. 286, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Experienced dyer and textile chemist, college graduate, 12 years' experience as dyer of yarn and piece goods. Rayon, cotton, wool and uniforms. Capable to improve and economize. Employed. Would go South. Write Box No. 287, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Chemist, B.S., M.S., age 41. Will promote sound chemical products or add capital to growing concern requiring services of capable executive with successful business record. Prefer Philadelphia, New York or vicinity. Write Box No. 288, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: One used Color Fadeometer. Type FDA or similar kind. Please quote price, condition, volts, amperes, and D.C. or cycles if A.C.. Write Box No. 289, American Dyestuff Reporter, 440 Fourth Ave., New York. N. Y.

POSITION WANTED: Executive and sales type of textile chemist and colorist desires position in sales, contact, promotional or supervisory work. Age 43, American, married. Factory, laboratory, office and purchasing experience. Textile school graduate. Good correspondent. Good personality. Accustomed to assuming responsibility and exercising initiative. 21 years' experience in the textile, chemical and dye industries. Opportunity most important. Now employed. Available on short notice. Write Box No. 290, Amercian Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Established firm of manufacturing chemists have territory open for their products. Concerns selling dyestuffs will find our line advantageous as an additional source of revenue. Write Box No. 291, American Dyesutff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Young man with good personality to sell and demonstrate to textile and paper mills. Knowledge of textile processing required. State age, education, experience, and salary expected. Write Box No. 292, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.